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# USSR Report

CHEMISTRY

No. 73

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## USSR REPORT

## CHEMISTRY

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## ADSORPTION PHENOMENA

UDC 541.183

THE TEMPERATURE DEPENDENCE OF ADSORPTION, GENERAL PRINCIPLES IN THE ADSORPTION OF INDIVIDUAL SUBSTANCES

Moscow IZVESTIYA AKADEMII NAUK SSSR--SERIYA KHMICHESKAYA in Russian No 4, 1980  
pp 759-765 manuscript received 7 Mar 79

KABANOVA, O. N., SERPINSKIY, V. V., YAKUBOV, T. S., Institute of Physical Chemistry of the USSR Academy of Sciences, Moscow

[Abstract] The temperature dependence of adsorption was subjected to thermodynamic analysis to predict the temperature behavior of adsorption systems. The concept of isoergs or lines of constant value of the free enthalpy of adsorption, was introduced. The ratio of the differential entropy of adsorption calculated from the level of liquid entropy to the thermal adsorption coefficient along any isoerg is greater than zero. The abscissa of the deflection point  $a_p$  of the adsorption isotherm is not dependent on temperature in coordinates  $\ln p - a$ , while the ordinate of the deflection point  $\ln p_p$  is linearly dependent on inverse temperature. A single isotherm is sufficient to find the parameters of this dependence. The thermal equation of adsorption, all parameters of which can be determined by a single adsorption isotherm, is proposed. The equation easily describes the adsorption equilibria in a very wide range of pressures, temperatures and surface coverages.

Figures 4; references 6: 4 Russian, 2 Western.

[185-6521]

AEROSOLS

UDC 541.182.2/.3:533.735

THE THERMAL DIFFUSION PHORESIS OF FINE AEROSOL PARTICLES IN THE COURSE OF PHASE TRANSITIONS ON THEIR SURFACE

Moscow KOLLOIDNYY ZHURNAL in Russian Vol 42, No 3, May-Jun 80 pp 439-444  
manuscript received 21 Oct 78

BERESNEV, S. A., CHERNYAK, V. G. and SUYETIN, P. Ye., Ural University

[Abstract] The movement of a fine particle in a binary gaseous mixture with temperature and concentration gradients was analyzed. In such a situation, both components are capable of undergoing phase conversions on the surface of these particles. The partial vapor pressure differs from the saturated pressures, so that, during its motion, the particle can change its dimensions and temperature. An analysis of this phenomenon indicated that phase transitions occurring on the surface of fine particles moving through a binary gas system definitely affect the rate of their diffusion phoresis. In situations of low vapor concentrations, the effect of particle movement on the thermal phoresis is negligible. However, the movement of a drop in a vapor proper could have an effect on the rate of thermal phoresis, changing it by as much as 40 percent, depending on the coefficient of mass accommodation. References 16: 11 Russian, 5 Western.  
[212-7813]

UDC 541.18.02.182

THE EFFECT OF AIR STREAM CONTACT WITH A GENERATOR ON THE DISPERSION OF CONDENSING AEROSOLS

Moscow KOLLOIDNYY ZHURNAL in Russian Vol 42, No 3, May-Jun 80 pp 533-536  
manuscript received 19 Feb 79

BELYAYEV, S. P., KIM, N. S., OGANESYAN, S. Kh. et al., Institute of Experimental Meteorology, Obinsk

[Abstract] The aim of this study was to investigate the composition of dispersed aerosols formed during a movement of the aerosol generator through the air. Two experimental set-ups were used, one modelling a 60 m/sec movement of the generator, and another a 250 m/sec movement. The experiments were performed using pyrotechnical

generators of ice forming aerosols of silver iodide. It was shown that increasing the velocity of the generator decreased the average size of the particles. This was explained in terms of an air dilution of the aerosol mixture at the moment when the aerosol particles were formed. Other properties of aerosols, such as optical transparency, aggregate stability in the atmosphere, physical-chemical properties, etc. also depend on the difference in the velocity of the generator and aerosol stream. Figures 2, references 9: 8 Russian, 1 Western.  
[212-7813]

## CHEMICAL INDUSTRY

### MINISTER ASSESSES CHEMICAL INDUSTRY SHORTFALLS

Moscow PRAVDA in Russian 25 May 80 p 1

[Interview with V. S. Fedorov, minister of Petroleum Refining and Petrochemical Industry: "Industry for All"]

[Excerpt] Workers in the chemical industry are celebrating their trade holiday today. What they have brought to the holiday is described by V. S. Fedorov, USSR Minister of Petroleum Refining and Petrochemical Industry, at the request of PRAVDA correspondent V. Shilov.

At the November 1979 Plenum of the CPSU Central Committee, Comrade L. I. Brezhnev voiced a number of serious criticisms concerning the work of the chemical sectors of industry. Enterprises and associations are therefore now striving to eliminate the noted shortcomings, to overcome the lag, and to successfully complete the tasks of the quinquennium. It must be said that this is being achieved in many respects. During the 4 months of 1980, above-plan shipments to consumers included hundreds of tons of motor fuels, 120,000 tons of nitrogen fertilizers, about 200,000 tires, more than 5,000 tons of paints and varnishes, and many different recreational and household products.

About 60 percent of the growth in chemical production is targeted to occur at new production facilities. The large Mazheykay Petroleum Refinery in the baltic has started operating. Facilities producing isoprene and caoutchouc in Nizhnekamsk and Novokuybyshevsk have been accepted for operation. A mineral fertilizer plant in the Belorussian "Belarus'kaliy" Potassium Association and a polyvinyl alcohol refinery in Nevinomysk as well as many other facilities have been constructed. The main task now is for the young collectives to raise the capacity of equipment more quickly to the designed level. The experience o. the leading enterprises should serve as an example.

Warm salutations from Comrade L. I. Brezhnev to the Kirishi "Kirishinefteorgsintez" Petroleum Organic Synthesis Association was published yesterday. The complex for the production of liquid paraffins here was brought up to the designed capacity 3 months ahead of schedule. The success is attributable to capable collaboration between builders and operators. Accounts were settled with contractors only after start-up which prodded the participants to accelerate the work pace. The collective,

which was formed earlier, actively participated in the start-up adjustment work of facilities. As a result, all installations here were put on stream ahead of schedule; and because of modernization of equipment, the capacity of the main technological units increased by 50 percent.

Many such examples may be cited. Some 26,000 workers in the chemical branches and 300 brigades, sections and shifts in petroleum refineries have pledged to fulfill the goals of the five-year plan ahead of schedule and to firmly observe their word.

The operations of the chemical branches of industry also have serious shortcomings. A number of enterprises do not meet orders in terms of product mix. Many new plants operate at less than full capacity, as the result of which the national economy fails to receive products worth millions of rubles. Consumer complaints concerning the quality of engine oils, tires, chemical fibers and paints are fully justified. The problems of capital construction are extremely acute. Although valid solutions have been proposed for all of these problems, they are often implemented with insufficient vigor and comprehensiveness. The first priority task of the ministerial apparatus of the production associations is to radically improve management, to strengthen the control over the implementation of designated measures, and to occupy itself more with direct organisational work.

We are observing our holiday essentially at the threshold of the Eleventh Five-Year Plan. Today's results are in large measure determined by the future pace of work. In the development of new plans, particular attention is devoted to areas in Siberia and the Far East, where a large base of raw materials is located. Erection of high-capacity refining and chemical complexes is already under way in Omsk, Tomsk, and in other large centers. Their construction and development will make it possible to reduce hauling of raw materials and to more fully satisfy the chemical needs of industry and agriculture there.

Consumer goods are the object of special attention. Presently, the output of consumer oriented chemistry will increase 15 percent, and the production of detergents will grow by more than 30 percent. The development of recreational and household products remains one of the principal goals of our work.

The plant for the production of isoprene caoutchouc at the Nizhnekamsk "Nizhnekamskneftkhim" Petrochemical Production Association has been put on stream. The enterprise has pledged to bring production up to designed capacity ahead of schedule.  
[01-P]

CSO: 1841

BRIEFS

WATER DECOMPOSED BY LIGHT--A new method for decomposition of water into hydrogen and oxygen has been developed in a department of the Institute of Chemical Physics of the USSR Academy of Sciences. The scientists have decided to bypass expensive electrolysis and to decompose water by bombarding it with photons of visible light. The idea was thought to be promising for a long time; however, an effective catalyst was not available. The catalysts have now been found. They are cobalt and ruthenium. Their water compounds--hydrocomplexes--have for the first time in practice assisted the photons to decompose water into hydrogen and oxygen in special three-chamber vessels. A mercury lamp with an interference light filter was used as the light source. [Text] [Moscow SOTSIALISTICHESKAYA INDUSTRIYA in Russian 21 Aug 80 p 4] [02-P]

PHOSPHORITE PLANT MINE--Bryansk. A large open-cut mine was put into operation at the Bryansk Phosphorite Plant. According to estimates of specialists, enough deposits exist for the production of mineral fertilizers to last many years. Having equipped the mine with new technology, the enterprise has pledged to bring the annual volume of ore extraction to 900,000 tons. [Text] [Moscow SEL'SKAYA ZHIZN' in Russian 13 Jul 80 p 1] [04-P]

CSO: 1841

ELECTROCHEMISTRY

UDC: 546.723-31

PRODUCTION OF ACTIVE IRON OXIDE FOR FERRITES

Kiev KHIMICHESKAYA TEKHNOLOGIYA in Russian No 1, Jan-Feb 80 pp 8-9

STETSENKO, V. I., LUKACHINA, Ye. N.

[Abstract] A study is presented of the physical and chemical properties of iron oxide synthesized from hydroxides under various conditions. The purpose of the study was to produce a highly active and pure hydroxide (oxide) of iron with reproducible properties to be used as a standard raw material in the manufacture of ferrites.  $\alpha$ -FeOOH was precipitated from a 2M solution of ferric nitrate, ferric sulfate and ferric chloride. The salt solutions were heated to 70°C then poured into a cold 25%  $\text{NH}_4\text{OH}$  solution or an 8n solution of NaOH with agitation. The sediment was then dried at 120°C (for ammonia precipitation) or 150°C (caustic soda precipitation). The use of active iron oxide obtained by the hydroxide method can increase the yield of usable ferrite cores with rectangular hysteresis loops by a factor of 1.5. References: 7 Russian.

[137-6508]

## FERTILIZERS

UDC 631.847"71"

### BASIC TRENDS OF DEVELOPMENT OF THE TECHNOLOGY OF CARBAMIDE PRODUCTION

Moscow KHMICHESKAYA PROMYSHLENNOST' in Russian No 5, 1980 signed to press 29 Apr 80 pp 282-285

GORLOVSKIY, D. M., KUCHERYAVYY, V. I., KOSHCHERENKOV, N. N., and ZINOV'YEVA, L. K.

[Abstract] Patent materials not discussed in a series of previously published articles concerning this subject (CHEMICAL INDUSTRY 1968, No 1, p 45; 1970, No 7, p 504; 1972, No 8, p 601; 1973, No 4, p 278; 1975, No 4, p 274; 1977, No 3, p 189) were reviewed briefly. Areas discussed include: the carbamide synthesis assembly, recuperation of unconverted reagents, reduction of energy expenditures, separation of carbamide from solution and improvement of the quality of its commercial forms and improvement of the quality of the product. A table of methods for improving the commercial properties of carbamide is presented. References: 30.  
[214-2791]

UDC 631.85:621.36

### ELECTROTHERMAL PRODUCTION OF FUSED DEFLOURINATED PHOSPHORUS FERTILIZERS

Moscow KHMICHESKAYA PROMYSHLENNOST' in Russian No 3, 1980 pp 157-159

MIKHAYLIN, A. D., KOSTYL'KOV, I. G., ZHIZIYEV, T., KLINITSKIY, A. I., TALANOV, N. D., LYAPUNOV, M. I. and USTYUGOVA, L. M.

[Abstract] A new technological process for the electrothermal production of fused defluorinated phosphate fertilizers and feed phosphate is examined in connection with the importance of producing long-acting phosphate fertilizers for nonchernozem soils from virtually all available phosphorus ores. The basic steps of the proposed process are examined and a schematic diagram of a commercial plant with a high-unit capacity furnace is presented. The process produces no effluents and minimal exhaust gases and completely solves problems of the production of defluorinated phosphates and hydrogen fluoride without expensive sulfuric and phosphoric acids. Figures 1; tables 1; references: 4 Russian.  
[158-7872]

UDC 631.895

## COMPLEX POLYMERIC FERTILIZERS

Moscow KHIMICHESKAYA PROMYSHLENNOST' in Russian No 6, 1980 pp 346-348

KUZNETSOVA, A. Ye., TARKHANOVA, L. S., KOGAY, S. N. and FEDOROVA, I. N.

[Abstract] Various techniques for the production of complex polymeric fertilizers (CPF) have recently been developed. The principal raw materials used to make CPF are a 70% solution of carbamide (following second-stage distillation, with an ammonia content of not more than 0.6-0.8 mass%) or solid carbamide, 37% formalin, ammophos in granulated or slurry form, and technical grade calcium chloride. CPF have proved their worth on salt-infested virgin lands, where they caused crop harvests to increase 5.1 to 12.5 centners per hectare. CPF are relatively resistant to the effect of moisture, which is important in Soviet Central Asia where average relative humidity is 6%. Thus, CPF, which contain urea-formaldehyde compounds and phosphorus-containing ingredients, are promising fertilizers for irrigated lands and in particular for regions with salt-infested soils. A pilot facility for the production of 1,500 tons of CPF annually has been recently designed. Figure 1; references 11: all Russian.

[209-1386]

UDC 546.171.1

## A STUDY OF AMMONIA SYNTHESIS IN THE PRESENCE OF URANIUM CATALYSTS

DOKLADY AKADEMII NAUK SSSR in Russian Vol 251, No 2, 1980 pp 375-379 manuscript received 6 Sep 79

SPITSYN, V. N., YEROFEYEV, B. V., MIKHAYLENKO, I. Ye., GORELKIN, I. I. and IVANOV, L. S., Moscow, Institute of Physical Chemistry, USSR Academy of Sciences; Moscow Institute of National Economy imeni G. V. Plekhanov

[Abstract] The effect of induced radioactivity and chemical composition of uranium catalysts on their catalytic activity in the ammonia synthesis reaction was investigated on specimens of 99.88% pure uranium metal measuring 2 x 3 x 0.2 mm and on chips, with the chips being first irradiated in a nuclear reactor in the presence of an integral slow neutron flux of  $4.3 \cdot 10^{16}$  n/cm<sup>2</sup>. Ammonia yield in a catalytic reactor was determined as a function of the formation time of uranium catalysts (uranium nitride) in the presence of a flow of nitrogen-hydrogen mixture at temperatures ranging from 380 to 405°C. It was found that the catalytic activity of the uranium catalysts thus formed is superior to the activity of industrial iron catalysts: the maximum ammonia yield in the presence of uranium catalysts is 32% higher. The optimal formation temperature of uranium catalysts should not exceed 400°C. This is because in uranium nitride at < 400°C the interaction process in the uranium-nitrogen system takes place with the participation of hydrogen through the formation of the hydride NH<sub>3</sub> which then interacts with nitrogen. At

>400°C hydrogen does not participate in the reaction and the directly interacting product has a dense structure resulting in the formation of a compact nitride film on the metal surface, which inhibits further interaction. As regards the effect of radiation on catalytic activity, it was found to be nugatory: irradiated and non-irradiated specimens of uranium catalysts displayed the same catalytic activity. On the whole, uranium catalysts are promising for use at comparatively low pressures, when their composition is close to that of uranium dinitride, whose catalytic activity is the highest. Figures 3; references 12: 5 Russian, 7 Western. [163-1386]

FOOD TECHNOLOGY

UDC 634.0.813.2

GLYCOPROTEINS OF PLANT TISSUE. I: ISOLATION OF BIRCH BARK GLYCOPROTEINS AND DETERMINATION OF THEIR CHEMICAL COMPOSITION

Riga KHIMIYA DREVESINY in Russian No 2, 1980 pp 100-103 manuscript received 11 May 79

SHISHKOVA, Z. P., IVANOVA, D. V., EGLE, V. B., GAYLITIS, Yu P. and SHMIT, U. Ya., Institute of Wood Chemistry, Latvian SSR Academy of Sciences

[Abstract] Carbohydrate-protein compounds of the glycoprotein type were experimentally isolated and analyzed. Of the methods tested, the most effective in isolating the glycoproteins was the Gladyshev method, consisting in the treatment of plant tissue with an urea solution in the presence of 1% NaOH. The isolated birch-bark glycoproteins were purified by dialysis with distilled water and gel-filtration on G-25 and G-50 sephadex compounds. The carbohydrate composition of the high-molecular part of the glycoproteins precipitated with  $\text{CCl}_3\text{COOH}$  or  $(\text{NH}_4)_2\text{SO}_4$  was determined by hydrolysis in soldered ampoules for 18 hr at 100°C. The principal ingredients of the glycoprotein sugars were found to be arabinose, xylose, and glucose. In the glycoproteins precipitated with ammonium sulfate the ratio of the carbohydrate part to the peptide part was 10:1, while in the glycoproteins precipitated with trichloroacetic acid that ratio was 20:1. In the glycoproteins precipitated with ammonium sulfate the principal amino acids are serine and aspartic acid, whereas in the glycoproteins precipitated with trichloroacetic acid the principal amino acids are aspartic and glutamic acids and proline. It may be assumed that in the isolated glycoproteins the relationship between the protein and carbohydrate parts is mediated by serine, asparagine, and oxyproline. Figure 1; references 10: 5 Russian, 5 Western.

[160-1386]

UDC 663.12:552.577:547.915

## MICROBIOLOGICAL SYNTHESIS OF LIPIDS ON HYDROLYTIC MEDIA

Moscow KHIMIYA TVERDOGO TOPLIVA in Russian No 2, 1980 pp 172-175 manuscript received 17 Jan 78

OBRAZTSOVA, N. V., MIKHEYEVA, L. D., YEVDOKIMOVA, G. A., RAYTSINA, G. I. and IVANOVICH, Z. A., Institute of Microbiology, Belorussian SSR Academy of Sciences

[Abstract] It is known that lipid-forming yeasts can be grown on sulfate hydrolysates of top peat. However, bottom peat is richer in humic acids, amino acids, and other biologically active substances. In this connection, the process of the growth and lipid-formation in yeasts grown on the hydrolysates of mixtures of top and bottom peat (I) as well as on wood hydrolysates was investigated. *Lipomyces lipoferus* 199 yeast was used as the lipid producer. The hydrolysates were neutralized to pH 5.5. Mineral salts were added in the amounts (g/liter):  $\text{KH}_2\text{PO}_4$ , 0.12;  $\text{MgSO}_4$ , 0.04;  $\text{CaCl}_2$ , 0.04. The yeast was cultured in rocking flasks at 23-25°C for days. The lipids were isolated by extraction of biomass by means of diethyl ether following treatment with 10% HCl. Gas-liquid chromatography revealed that in the yeast lipids produced on wood hydrolysates the content of unsaturated fatty acids was higher (71.9%) than in the lipids produced on peat hydrolysates (58.6-65.8%). The economic and fats coefficients (output in grams per 100 g sugar used) for *L. lipoferus* 199 yeast produced on hydrolysates of top peat are virtually the same as those for the mixtures of hydrolysates of top and bottom or sedge peat. As for wood hydrolysates, their output of yeast lipids was somewhat lower, owing to a lower production of biomass. References 9: 8 Russian, 1 Western.

[147-1386]

UDC 636.086.25:661.3

## AMMONIZATION OF STRAW

Moscow KHIMIYA V SEL'SKOM KHOZYAYSTVE in Russian No 11, 1979 pp 38-40

ZAFREN, C. Ya., Professor, All-Union Scientific Research Institute of Fodder

[Abstract] A process is explained, whereby straw is treated with ammonia water and liquid ammonia to improve the digestibility of the cellulose and lignin contained in straw, its total nutritional value and its protein value, so that it can be used as a feed supplement for cattle and sheep. The process increases the food value of straw from 0.2 to 0.5 feed units. The straw is covered with 16 x 16 m airtight polyethylene film canopies to retain the volatile ammonia, with which the straw is irrigated through perforated pipes, and the straw is left covered for 4-5 days in warm weather and 2-3 weeks in cold weather. The product is ready for consumption after the ammonia odor disappears and can be stored all winter long. It may be added to silage without an appreciable loss of nutritional value. Bull calves fed on a diet of 25 kg of corn-straw silage and 2 kg of concentrates gained a mean daily weight increment of 1,014 g, compared with 804 g for silage with raw straw.

[154-7872]

UDC 636.085.54:661.717.5

UREA CONCENTRATE -- AN IMPORTANT RESERVE FOR INCREASING THE EFFECTIVENESS OF LIVESTOCK FEEDING

Moscow KHIMIYA V SEL'SKOM KHOZYAYSTVE in Russian No 11, 1979 pp 30-34

AYBETOV, A. V., candidate of agricultural sciences, Northwest Scientific Research Institute of the Dairy and Grazing Industry

[Abstract] Tests that demonstrate the benefit of using urea concentrate in livestock feed are described. A new method is explained, whereby urea concentrate is produced by cooking under pressure a mixture of grain and urea in continuous-action extruders without adding sodium bentonite plasticizer. The resulting concentrate is slowly hydrolyzed in the rumen at the rate of decomposition of starch so that bacteria can utilize all of the ammonia for synthesizing growth protein. The concentrate tastes better, is more easily digested and has a higher protein equivalent than the previously utilized feed additive. Any plant with access to grain, urea and an extruder can manufacture the less expensive new product because no plasticizers and the associated machinery for making them are needed. The concentrate can also be used in wet feed mixtures. Figures 1; tables 5.

[154-7872]

UDC 58/192, 58/198

MICROBIAL METABOLITES AND INCREASE IN CROP YIELDS

Alma-Ata VESTNIK AKADEMIJ NAUK KAZAKHSKOY SSR in Russian No 2, 1980 pp 27-30

TULEMISOVA, K. A., KULDYBAYEV, M. M. and MAZUNINA, V. I.

[Abstract] A long term study shows that certain actinomycetic strains and their metabolites stimulate the growth and development of crops. Metabolites of *Actinomyces griseoruber* strain 1618, *Actinomyces fumanus* strain 1389, and the antibiotic A-23/791 diluted 1:1000 in alcohol were applied in field and laboratory experiments to soybeans, corn, sugar beets, rice, cucumbers and cabbage. Treatment of Strain 1389 with metabolites resulted in soybean yields that were 16.4 centners per hectare higher than the controls. Similarly, it increased corn yields by 16-18 percent. The antibiotic A-23/791, when applied to the cucumber crop reduced by 95% the root rot of cucumbers and increased their yields by 30-70%. The antibiotic 1618-306, synthesized from Strain 1618 which consists of 7 components belonging in the anthracycline and anthracyclinone groups, has components (A, A<sub>1</sub>, B, C) which proved to stimulate the germination rate of cabbage, rice, and sugar beet seeds. Thus, preplanting treatment of crop seeds with metabolism products of *actinomyces* 1618, 1389, and A-23/791 stimulates the growth and development of crops and enhances their yields. References 10: 8 Russian, 2 Western.

[191-1386]

## ORGANOPHOSPHORUS COMPOUNDS

UDC 542.91:547.1'118

### NUCLEOPHILIC ADDITION OF AMINES TO VINYLIDENE DIPHOSPHONIC ACID

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 5, 1980  
pp 1211-1212 manuscript received 21 Jan 80

ALFER'YEV, I. S., KOMLYAREVSKIY, I. L., MIKHALIN, N. V., NOVIKOVA, V. M.,  
Institute of Chemical Kinetics and Combustion of the Siberian Department, USSR  
Academy of Sciences, Novosibirsk

[Abstract] Vinylidene diphosphonic acid and its salts can be produced by thermal dehydration of the salts of easily available 1-oxyethylidene-1,1-diphosphonic acid. Vinylidene diphosphonic acid and its salts are capable of nucleophilic addition of amines of different classes and N-substituted 2-aminoethylidene-1,1-diphosphonic acids are formed. Numerous  $\beta$ -amino-derivatives of ethylidene-1,1-diphosphonic acid of the aliphatic, aromatic and heterocyclic series were synthesized by using the described method. References: 2 Western.  
[190-6521]

UDC 542.941:542.955:547.371:546.185'131

### REDUCTION OF PHOSPHORUS PENTACHLORIDE ADDUCTS CONTAINING ALKENES BY ARSINE

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 5, 1980  
p 1208 manuscript received 3 Mar 80

KROLEVETS, A. A., FOKIN, A. V.

[Abstract] White phosphorus,  $\text{PH}_3$  and other compounds used for reduction of phosphorus pentachloride adducts with alkenes to corresponding dichlorophosphines are effective only at  $-5^\circ$  and above.  $\text{AsH}_3$ , which is effective even at  $-20^\circ$ , can be used as a soft reduction agent in the adduct reduction process. This permits the use of  $\text{AsH}_3$  to establish the structure of low-stable adducts of phosphorus pentachloride with alkenes. The yield of dichlorophosphines comprises 85-90 percent. References: 2 Russian.  
[190-6521]

UDC 542.91:547.1'118

2-SUBSTITUTED 1,3-DIBENZYL-1,3,2-DIAZOPHOSPHALANES

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 5, 1980  
pp 1183-1185 manuscript received 16 Nov 79

PUDOVIK, M. A., MUSLIMOVA, N. A., PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov of the Kazan' Branch, USSR Academy of Sciences

[Abstract] The reactions of *N,N'*-dibenzylethylenediamine with some derivatives of trivalent phosphorus were studied with respect to synthesis of five-membered phosphorus-, nitrogen- and oxygen-containing heterocycles. The interaction of *N,N'*-dibenzylethylenediamine with  $\text{PCl}_3$  in the presence of a base leads to formation of 2-chloro-1,3-dibenzyl-1,3,2-diazophospholane. A series of 2-substituted 1,3-dibenzyl-1,3,2-diazophosphalanes was produced through interaction of phosphorus trichloride, hexaethyltriaminophosphine and tetraethylamidoalkylphosphites with *N,N'*-dibenzylethylenediamine. References: 1 Russian.

[190-6521]

UDC 542.91:541.69:547.1'118

SYNTHESIS, INTERACTION WITH ESTERASES AND TOXICITY OF O-ALKYL-S-[ (CARBOALKOXY-METHYL)MERCAPTO]METHYLPHOSPHONOTHIOATES

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 5, 1980  
pp 1131-1136 manuscript received 16 Apr 79

MASTRYUKOVA, T. A., SHIPOV, A. E., YEMKUZHEVA, Z. K., BRESTKIN, A. P., BRIK, I. L., MANDEL'SHTAM, Yu. E., FEDIN, A. N., NOZHKO, T. I., TILYABAYEV, Z., ROSLAVTSEVA, S. A., KAGAN, Yu. S., YERSHOVA, Ye. A., KABACHNIK, M. I., Institute of Elementary Organic Compounds of the USSR Academy of Sciences, Moscow

[Abstract] The effect of R and R' substitutes on the capability of monothio-analogs of  $\text{CH}_3(\text{RO})\text{P}(\text{S})\text{S}(\text{CH}_2)_n\text{SCH}_2\text{COOR}'$  compounds to inhibit the esterases of mammals and arthropods was traced and the data were compared to the toxicity of the substances. The degree to which the capability of methylthiophosphonates to inhibit the cholinesterases of mammals is applicable to the esterases of arthropods was determined. A series of O-alkyl-S-[ (carboalkoxymethylmercapto)methyl] methylthiophosphinates was produced by reaction of sodium salts of methylthiophosphonic acids with halogen derivatives. The rate of interaction with esterases of mammals and arthropods increases with an increase of the alkyl in the alkoxylic group of the phosphorous atom for the compounds produced. The toxicity of the compounds to arthropods decreases. Figures 1; references 9: 7 Russian, 2 Western.

[190-6521]

UDC 542.91:541.6:547.1'118

SYNTHESIS AND DETERMINATION OF THE ABSOLUTE CONFIGURATION OF R- $\alpha$ -AMINOBENZYL-PHENYLPHOSPHINE ACID

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 5, 1980  
pp 1125-1131 manuscript received 10 Apr 79

BELOV, Yu. P., RAKHNOVICH, O. B., DAVANKOV, V. A., GODOVIKOV, N. N.,  
ALEKSEANDROV, O. G., STRUCHKOV, Yu. T., Institute of Elementary Organic Compounds  
of the USSR Academy of Sciences, Moscow

[Abstract] Ethyl- $\alpha$ -aminobenzylphenylphosphinate was synthesized and the absolute configuration of its C(R),P(S)-isomer was established. R- $\alpha$ -aminobenzylphenylphosphinic acid was produced and asymmetrical ion exchange resins with groupings of C(R),P(S)-EABPP and of R- $\alpha$ -aminobenzylphenylphosphinic acid were synthesized. A method of producing ethyl- $\alpha$ -aminobenzylphenylphosphinate from hydrobenzamide and ethylphenylphosphinate is proposed. The stereospecific nature of this reaction in the ratio of configurations of C and P atoms was discovered.  $\alpha$ -Aminobenzylphenylphosphinic acid and its ethyl ester were produced in optically active form and the absolute configurations of asymmetric C and P atoms were determined. The kinetics of hydrolysis of ethyl- $\alpha$ -aminobenzylphenylphosphinate by dry HBr was studied. Polystyrene type absorbents with groupings of R- $\alpha$ -aminobenzylphenylphosphinic acid and its ethyl ester were produced for the first time. References 19: 13 Russian, 6 Western.  
[190-6521]

UDC 542.91:547.1'118

INTERACTION OF DIMETHYLPHOSPHOROUS ACID WITH 2,5-BIS(CARBOMETHOXY)-3,4-DIPHENYLCYCLOPENTADIENONE

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 5, 1980  
pp 1119-1125 manuscript received 24 Jan 79

ARBUZOV, B. A., PUZHENKOVA, A. V., GALYAUTDINOV, N. I., SHAYKHULLINA, R. F.,  
Chemical Institute imeni A. M. Butlerov of Kazan' State University imeni V. I.  
Ul'yanov-Lenin

[Abstract] The interaction of dimethylphosphite with 2,5-bis(carbomethoxy)-3,4-diphenylcyclopentadienone (BCDC) was studied. BCDC interacts with dimethylphosphite both in the presence of triethylamine and in the absence of catalysts with formation of dimethyl esters of 2-oxo-4,5-diphenyl-1,3-dicarbomethoxy-4-cyclopentene- and 2-oxo-4,5-diphenyl-1,3-dicarbomethoxy-3-cyclopentene-1-phosphoric acids. BCDC does not form products of 1,4-addition with dimethylphosphite in the absence of catalysts as does tetraphenylcyclopentadienone. Addition products for the carbonyl group of  $\alpha$ -oxyphosphonate and enolphosphate are also not found in bases as is observed for tetraphenylcyclopentadienone. References 17: 8 Russian, 9 Western.  
[190-6521]

UDC 632.954

TETRACHLORANHYDRIDES OF BISACYLAMIDOPHOSPHORIC AND DICHLOROANHYDRIDES  
(BISACYLAMIDOALKYL (ARYL)) PHOSPHONIC ACIDS

Kiev KHIMICHESKAYA TEKHNOLOGIYA in Russian No 3, 1980 signed to press 7 May 1980,  
pp 53-55

RUDAVSKIY, V. P.

[Abstract] Biological activity of phosphorylated derivates of dicarbonic acids was studied and discussed. Two methods of production of tetrachloranhydrides of bisacylamidophosphoric and dichloroanhydrides of (bisacylamidoalkyl (aryl)) phosphonic acids were described and findings were presented in two tables and discussed. References 3.

[211-2791]

UDC 542.91:547.1'127'118

SYNTHESIS OF S-(*o*-CARBORAN-1-YLMETHYL)THIOPHOSPHONATES

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 6, Jun 80  
pp 1455-1456 manuscript received 11 Jan 80

KABACHNIK, M. I., GODOVIKOV, N. N. and RYS, Ye. O., Institute of Metal Organic Compounds, USSR Academy of Sciences, Moscow

[Abstract] Salts of *o*-ethylmethyl- or *o*-ethylphenylthiophosphoric acid react with propargyl chloride in ethanol to yield *O*-ethyl-S-propargylmethythiophosphonate, b.p. 87-90°C/1 mm,  $n_D^{22}$  1.5010 and *O*-ethyl-S-propargylphenylthiophosphonate, b.p. 124-126°C/1 mm,  $n_D^{24}$  1.5615. In the presence of dimethyl aniline in toluene, these products react with decaborane to yield highly effective inhibitors of cholinesterase. These inhibitors are *O*-ethyl-S-(*o*-carboran-1-ylmethyl)methylthiophosphonate, m.p. 82-84°C and *O*-ethyl-S-(*o*-carboran-1-ylmethyl)-phenylthiophosphonate, m.p. 61-62°C. No references.

[210-7813]

UDC 547.1'13:541.49

TETRAKIS(TRIPHENYLPHOSPHINEGOLD)AMMONIUM

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 6, Jun 80  
p 1455 manuscript received 7 Mar 80

PEREVALOVA, E.G., SMYSLOVA, Ye. I., DYACHENKO, V. P. et al., Institute of Metal Organic Compounds, USSR Academy of Sciences, Moscow, Moscow State University imeni M. V. Lomonosov

[Abstract] Liquid ammonia reacted with  $(Ph_3PAu)_3O^+BF_4^-$  in THF at  $-60^\circ C$  to yield tetrakis(triphenylphosphinegold)ammonium borofluoride, decomposition point  $242-244^\circ C$ . The product is soluble in  $CH_2Cl_2$  and  $CHCl_3$ . It can be split with 4 moles of  $HCl$  to yield  $AuClPF_3$  and with  $(CH_3)_2CO$  to yield  $Ph_3PAuCH_2COCH_3$ . References: 2 Russian.  
[210-7813]

UDC 542.91:547.558.1'161

### STYRYLTETRAFLUOROPHOSPHORUS

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 6, Jun 80  
p 1454 manuscript received 22 Feb 80

KROLEVETS, A. A., KOLOMIYETS, A. F. and FOKIN, A. V.

[Abstract] 35.6 g  $SbF_3$  was added at  $15-20^\circ C$  to a benzene solution of styryltetra-chlorophosphorus obtained from 31.2 g  $PCl_5$  and 15.6 g styrene in 100 ml benzene. The mixture was kept for 2 hrs at  $20^\circ C$  and then for 4 hrs at  $30^\circ C$ . It was then distilled to yield 20.5 g (65 percent) of styryltetrafluorophosphorus (I), b.o.  $61-63^\circ C/1.5$  mm,  $d_4^{20} 1.4950$   $n_D^{20} 1.5260$ . With  $Ac_2O$ , I gave styrylphosphonic acid difluoride, b.p.  $114-115^\circ C$  (10 mm)  $n_D^{20} 1.5140$ ,  $d_4^{20} 1.3360$ . References: 2 Western.  
[210-7813]

UDC 542.91:547.1'118

### 1-OXO-1-( $\beta$ -CHLOROETHOXY)-2-METHYLPHOSPHACYCLOPENTENE-3

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 6, Jun 80  
pp 1453-1454 manuscript received 6 Feb 80

KROLEVETS, A. A., KOLOMIYETS, A. F. and FOKIN, A. V.

[Abstract] To a cooled suspension ( $-30^\circ C$  to  $-25^\circ C$ ) of 41.6 g  $PCl_5$  in a 10:1 mixture of toluene:MeCN, 13.6 g piperylene was added, while stirring. The mixture was kept at  $-30^\circ C$  for 3 hrs, then 26.4 g dry ethylene oxide was bubbled through it. After fractional distillation, 13 g of 1-oxo-1-( $\beta$ -chloroethoxy) 2-methylphosphacyclopentene-3, b.p.  $68-70^\circ C/0.001$  mm was isolated,  $n_D^{20} 1.4865$ ,  $d_4^{20} 1.3713$ . No references.  
[210-7813]

UDC 542.91:547.1'118

REACTION OF DIALKYLCHLORO- AND ALKYLDICHLOROPHOSPHITES WITH N,N'-DIBUTYL-2,3-BUTANEDIIMINE

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 6, Jun 80  
p 1452 manuscript received 4 Feb 80

KIBARDIN, A. M., GAZIZOV, T. Kh. and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch, USSR Academy of Sciences

[Abstract] Diethylchlorophosphate reacts at 20°C with N,N'-dibutyl-2,3-butanediimine in pentane, forming 2-ethoxy-2-oxo-1,3-dibutyl-4,5-dimethyl-1,3,2-diazaphospholene at a yield of 42.3 percent, b.p. 114°C/0.1 mm,  $d_4^{20}$  1.0125,  $n_D^{20}$  1.4810. In the case of ethyldichlorophosphate, the product is 2-chloro-2-oxo-1,3-dibutyl-4,5-dimethyl-1,3,2-diazaphospholene (the yield - 71.5 percent), b.p. 111°C/0.06 mm,  $d_4^{20}$  1.0958,  $n_D^{20}$  1.5032. No references.  
[210-7813]

UDC 542.92:547.1'118

THERMOLYSIS OF TETRAMETHOXY(DIMETHOXYMETHYL)PHOSPHORANE

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 6, Jun 80  
pp 1451-1452 manuscript received 2 Jan 80

NESTEROV, L. V. and KREPYCHEVA, N. Ye., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch, USSR Academy of Sciences

[Abstract] Tetramethoxy(dimethoxymethyl)phosphorane breaks down when heated to 125°C in a sealed tube to yield trimethylphosphite and trimethylorthoformate. The products distill over as a mixture at 100-109°C with an 80 percent yield. Bicyclic bis(ethylenedioxy)(dimethoxymethyl)phosphorane remains stable even on prolonged heating to 200°C. References 5: 2 Russian, 3 Western.  
[210-7813]

UDC 542.91:547.1'118

TETRAALKOXY(DIMETHOXYMETHYL)PHOSPHORANES

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 6, Jun 80  
p 1450 manuscript received 26 Nov 79

NESTEROV, L. V. and ARBUZOV, N. Ye., Kazan' Branch, USSR Academy of Sciences

[Abstract] Addition of 1-2 parts of  $\text{CHCl}_3$  to a cooled (25°C) energetically stirred suspension of 4 parts of  $\text{MeONa}$  in hexane solution and 1 part of trimethylphosphite

yields tetramethoxy(dimethoxymethyl)phosphorane (35 percent), b.p. 49-50°C/0.2 mm,  $d_4^{20}$  1.1430,  $n_D^{20}$  1.4308. Replacement of trimethylphosphite with methylethylene-phosphite yields dimethoxy(ethylenedioxy)-(dimethoxymethyl)phosphorane in 13 percent, b.p. 79-81°C/0.2 mm,  $d_4^{20}$  1.2381,  $n_D^{20}$  1.4535. References 2: 1 Russian, 1 Western.  
[210-7813]

UDC 542.91:547.455:547.1'118

#### SYNTHESIS OF 3,5,6-BICYCLOPHOSPHITES OF 1,2-O-ISOPROPYLIDENE-5(6)-MERCAPTO-5(6)-DESOXY- $\alpha$ -D-GLUCOFURANOSE

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 6, Jun 80  
pp 1441-1442 manuscript received 10 Dec 79

NIFANT'YEV, E. Ye., KOROTEYEV, M. P., LYSENKO, S. A., et al., Institute of Organic Chemistry imeni N. D. Zelinskyy, USSR Academy of Sciences Moscow, and Moscow State Pedagogical Institute imeni V. I. Lenin

[Abstract] 6-Mercapto-6-desoxy- and 5-mercaptop-5-desoxy-1,2-O-isopropylidene- $\alpha$ -D-glucofuranose can be easily phosphorylated with hexamethyltriamidophosphite yielding corresponding 3,5,6-bicyclothiophosphites, which, in contrast to other thiophosphites, do not oxidize nor do they add sulfur. They can be chlorinated rather easily, yielding (in case of the 5-mercaptop starting material) 3,5-cyclo-chlorophosphates of 1,2-O-isopropylidene-5-mercaptop-5-desoxy-6-chloro-6-desoxy- $\alpha$ -D-glucofuranose. References 6: 3 Russian, 3 Western.  
[210-7813]

UDC 542.91:547.1'118

#### THE REACTION OF PHOSPHORUS DITHIO ACIDS WITH THIONYL AMINES

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 6, Jun 80  
pp 1402-1404 manuscript received 18 Sep 79

KUTYREV, G. A., MAVRIN, V. Yu., CHERKASOV, R. A. et al., Kazan' State University imeni V. I. Ul'yanov-Lenin

[Abstract] The reaction of N-methyl- or N-phenylthionyl amine with three equivalents of O,O-diisopropyl- or O,O-dimethyldithiophosphoric acid in hexane leads to the formation of the aniline salt of dithiophosphoric acid and bis(O,O-dialkylthiophosphoryl)-disulfide. Dimethyl- and diisopropylthiophosphates react with N-acetylthionylamine forming acetamide and bis(thiophosphoryl)-disulfide. The reaction of benzoyl isocyanate with dimethylthiophosphoric acid yields N-benzoyl-S-(O,O-dimethylthiophosphoryl)-thiocarbamate. Thus it was shown that, in contrast to

isocyanates, thioureas can react with dithiophosphoric acid and yield bis(thiophosphoryl)-disulfides and a respective amine derivative, regardless of the substituent at the nitrogen atom. References 5: 4 Russian, 1 Western.  
[210-7813]

UDC 541.632:547.586.2

#### PHENYLALANINE DERIVATIVES CONTAINING THE ACYL RESIDUE OF THE STEREOISOMERIC N-(DIETHYLENIMIDO THIOPHOSPHORYL)-4-AMINOCYCLOHEXYLACETIC ACIDS

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHIMICHESKAYA in Russian No 6, Jun 80  
pp 1426-1428 manuscript received 19 Nov 79

PATOTSKENE, L. A., KARPAVICHYUS, K. I. and KNUNYANTS, I. L., Institute of Metal Organic Compounds, USSR Academy of Sciences Moscow, Institute of Biochemistry, Lithuanian SSR Academy of Sciences, Vilnius

[Abstract] Cis- and trans-4-aminocyclohexylacetic acids (ACHAA) were converted to carbobenzoxy derivatives, which upon reaction with p-nitrophenol yielded p-nitrophenyl esters of cis- and trans-4-ACHAA. These products were then condensed with ethyl ester of DL-phenylalanine yielding ethyl esters of carbobenzoxy-cis- and trans-4-aminocyclohexylacetyl-DL-phenylalanine. Removal of the protective carbobenzoxy groups followed by reaction with NH<sub>3</sub> in CHCl<sub>3</sub> yielded ethyl esters of cis- and trans-4-aminocyclohexylacetyl-DL-phenylalanine, which upon thiophosphorylation and condensation with ethylenimine produced ethyl esters of N-(diethylenimido thiophosphoryl)-cis- and trans-4-aminocyclohexylacetyl-DL-phenylalanine. Their soluble Na salts were obtained by alkaline hydrolysis. References: 2 Russian.  
[210-7813]

UDC 547.279.8'118:541.632

#### STEREORECHEMISTRY OF THE ALKYLATION OF OPTICALLY ACTIVE SELENOPHOSPHONIC ACIDS

Leningrad ZHURNAL OBUHCHEY KHIMII in Russian No 6, 1980 pp 1429-1430 manuscript received 1 Oct 79

NURETDINOV, I. A., BAYANDINA, Ye. V. and SADKOVA, D. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] The acylation of optically active selenophosphonic acids in the presence of bases results in the formation of selenophosphonic and carboxylic acid anhydrides. In this connection, reactions between optically active selenophosphonic acids (I) and various alkylating reagents were investigated, and it was established that in such cases the reaction products are optically active alkylselenium ethers of alkylphosphonic acids. The addition of triethylamine to solution of (-)-acid (I)

in diethyl ether results in the formation of salt (II) which reacts with  $\text{CH}_3\text{Cl}$  on heating of reaction mixture to 35°C (method a). Following removal of HCl base residue and vacuum distillation, a satisfactory yield of (-)-O-ethyl-Se-methyl-selenolphosphonate (IIIa) could be obtained. Heating of a mixture of trialkyl-phosphite and (+)-acid (I) to 120-130°C results in the formation of O-ethyl-Se-ethylethylselenolphosphonate (IIIb) and diethylphosphite (method b). The structure of all the substances synthesized was corroborated by IR and NMR  $^1\text{H}$  and  $^{31}\text{P}$  spectra.  
References: 2 Russian.  
[207-1386]

UDC 547.241+661.729

#### REACTION BETWEEN DIMETHYL-2-CHLOROETHYLPHOSPHONATE AND SALTS OF TERT.-BUTYL HYDROPEROXIDE

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 6, 1980 pp 1428-1429 manuscript received 28 Sep 79

BREL', A. K., BOGDANOVA, O. S. and RAKHIMOV, A. I., Volgograd Polytechnical Institute

[Abstract] Published literature lacks information on reactions between dimethyl-2-chloroethylphosphonate (I) and hydroperoxides and their salts. It is known that dialkylvinylphosphonate, derived from phosphonate (I), enters into a reaction with a large excess of hydroperoxide, to form dialkyl-2-tert.-butylperoxyethylphosphonates. It is now established that, depending on the conditions of the reaction of phosphonate (I) with Na- or K-salts of ter.-butyl hydroperoxide (II), either the halogen gets replaced by the ter.-butylperoxy group, with formation of dimethyl-2-tert.-butylperoxyethylphosphonate (III) (reaction in aqueous medium), or partial dehydrohalogenation takes place (reaction based on use of anhydrous salt of (II)). The yield of peroxide (III) is markedly influenced by reaction temperature and reactant ratio. Procedure: 0.15 mol of phosphonate (I) was added to 0.17 mol of 20-30% aqueous solution of salt (II), or droplets of 20-25% solution of 0.18 mol of NaOH(KOH) were added to a mixture of 0.17 mol tert.-butyl hydroperoxide and 0.15 mol of compound (I). The temperature of the reaction mass rose to 50-55°C and then slowly decreased. The mixture was then heated at 40-45°C for 1-1.5 hr, the organic layer was skimmed off, and the residue was neutralized with a HCl solution, dried, and vacuum-distilled. The thus synthesized peroxide (III) (yield 58%) was soluble in water and in polar organic solvents. Thermal decomposition of peroxide (III) in chlorobenzene solution (0.01 mol/liter) at 145, 150, 155, and 160°C revealed that the decomposition kinetics can be described by a first-order equation for up to 80% transformation. References: 1 Russian.  
[207-1386]

UDC 547.371.110:541.632

## SYNTHESIS OF OPTICALLY ACTIVE VINYL ESTERS OF ALKYLPHOSPHONIC ACIDS

Leningrad ZHURNAL OBSCHEY KHIMII in Russian No 6, 1980 pp 1423-1424 manuscript received 1 Oct 79

NURETDINOV, I. A., BUINA, N. A. and SIBGATULLINA, F. G., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] It was established that reactions between chiral O-alkyl-O-trimethylsilylalkylphosphonites and chloral result in the formation of optically active O-alkyl-(1-trimethylsiloxy-2,2,2-trichloroethyl)alkylphosphinates which on heating turn into O-alkyl-O-(2,2-dichlorovinyl)alkylphosphonates with segregation of  $(\text{CH}_3)_3\text{SiCl}$ . The products thus synthesized were O-ethyl-(1-trimethylsiloxy-2,2,2-trichloroethyl)-ethylphosphinate (IV) which on heating at 15-20 mm Hg and 100°C for 2 hr separates into  $(\text{CH}_3)_3\text{SiCl}$  and O-ethyl-O-(2,2-dichlorovinyl)-ethylphosphonate (V). These findings contribute to knowledge of chiral vinyl ethers of the acids of phosphorus. References: 4 Russian.

[207-1386]

UDC 547.78

## ON THE SYNTHESIS OF ALKYLAMIDOPYROPHOSPHATES

Leningrad ZHURNAL OBSCHEY KHIMII in Russian No 6, 1980 p 1422 manuscript received 25 Dec 79

ZHELTONOG, N. G., RATNIKOVA, T. V. and GINAK, A. I., Leningrad Technological Institute imeni Lensovet

[Abstract] Considering that methods for the synthesis of pyrophosphates with amido groups at the phosphorus atom are relatively uninvestigated, alkylamidopyrophosphates (I-III) were synthesized by reacting the corresponding tetraalkyldiamido-chlorophosphates with sodium sulfide (reactant ratio 3:1, dimethylformamide medium). 60 g of N,N-tetraethyldiaminochlorophosphate dissolved in 20 cc dimethylformamide were added to 3.18 g of sodium sulfide dissolved in 150 cc of dry dimethylformamide. The reaction mixture was thoroughly stirred for 6 hr at 100°C, the sodium chloride residue separated, and the solvent driven off. Yield: 85%. The alkylamidopyrophosphates thus synthesized were identified by IR spectral analysis and element analysis (P, N, S) to be: N,N-octapropyltetraamidopyrophosphate and N,N-octabutyl-tetraamidothiopyrophosphate. They are thick oily substances of dark-brown color. References: 8 Russian.

[207-1386]

UDC 547.26'118+547.811

## 2-PHOSPHORYLATED TETRAHYDROPYRANES

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 6, 1980 p 1421 manuscript  
received 29 Dec 79

GAZIZOV, M. B., RAZUMOV, A. I. and GIZATULLINA, I. Kh., Kazan' Chemico-  
Technological Institute imeni S. M. Kirov

[Abstract] It was found that under nonrigorous conditions, acid esters of tri-coordinated phosphorus react with 2-chlorotetrahydropyran in two directions, i.e. nucleophilic substitution of chlorine in accordance with the Arbuzov reaction (a) is also accompanied by the elimination of hydrogen chloride (b). Direction (a) is the principal one. In the case of esters of phosphorous and alkylphosphonous acids the scale of direction (b) increases with increase in the volume of substituents at the tricoordinated phosphorus acid. Only in the case of trimethylphosphite 0,0-diethyl-4-chlorophenylphosphonite the reaction proceeded solely in direction (a). The structure and composition of the reaction products in both directions were demonstrated by IR and NMR  $^1\text{H}$  spectra. Various diastereoisomers of these reaction products with two directly bonded asymmetrical P and C atoms are readily detected by the NMR method on  $^{31}\text{P}$  nuclei.

[207-1386]

UDC 547.241

## FORMATION OF A NEW TYPE OF 1,2-OXAPHOSPHOLENES IN THE REACTION BETWEEN HYPOPHOSPHOROUS ACID AND UNSATURATED ALDEHYDES

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 6, 1980 pp 1419-1421 manuscript  
received 25 Dec 79

YUDELEVICH, V. I., SOKOLOV, L. B., IONIN, B. I. and PETROV, A. A., All-Union Scientific Research Technological Institute of Antibiotics and Medical Enzymes Leningrad Technological Institute imeni Lensovet

[Abstract] Various methods exist for the synthesis of 1,2-oxaphospholenes. Analogous heterocycles are known to form upon interaction of phenylphosphonous acid with unsaturated ketones. It was established that aryl(alkyl)phosphonous acids (I) react with  $\alpha$ ,  $\beta$ -unsaturated aldehydes (II) to form 1,2-oxaphosphol-3-enes (III). As part of a continuing investigation of the reactivity of hypophosphites, hypophosphorous acid (IV) was successfully reacted with the same carbonyl compounds (II). 1,2-oxaphosphol-3-enes with exocyclic fragments containing unsaturated  $\text{C}=\text{C}$  bonds (V) were synthesized for the first time. Hypophosphorous acid, which contains two mobile hydrogen atoms at the phosphorus atom, reacts with two molecules of aldehyde, with subsequent cyclization of the intermediate phosphinic acid. This is accompanied by isomerization, with transfer of the double bond to position 3 of the cycle. These findings demonstrate the general applicability of intramolecular cyclization to compounds with the hydrophosphoryl group  $\text{H}-\text{P}(\text{O})\text{OH}$  in the reaction

with  $\alpha$ ,  $\beta$ -unsaturated aldehydes (II), which provides a simple and convenient technique for the synthesis of a new type of 1,2-oxaphosphol-3-enes with various exocyclic fragments having the phosphorus-carbon bond. References 3: 2 Russian, 1 Western.  
[207-1386]

UDC 547.241

#### INTERACTION OF DIETHYLPHOSPHONOPHENYLKETENE WITH HYRAZOIC ACID AND DIAZOMETHANE

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1418-1419 manuscript received 5 Nov 79

KOLODYAZHNYY, O. I., YAKOVLEV, V. N. and KUKHAR', V. P., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] Phosphorylated ketones are new and extremely reactive compounds which enter into the most varied transformations. In this connection, it was found that reacting diethylphosphonophenylketene (I) with hydrazoic acid results in the formation of phosphorus-containing isocyanate, while reacting (I) with diazomethane results in the formation of phosphorylated cyclobutanones. Ketene (I) readily combines with hydrazoic acid in ether solution at 5-10°C, yielding alpha-diethylphosphonophenylacetic acid azide (II). On heating to 80°C azide (II) releases nitrogen and turns into diethylphosphonobenzylisocyanate. Similarly, ketene (I) readily combines with diazomethane in ether at -20 to -40°C, with release of nitrogen. When the reactant ratio is 1:2, isomeric cyclobutanones (V, VI) appear (yield 50%). When the reactant ratio is 1:1, this results in a mixture of compounds which IR spectral analysis reveals to consist of unreacted ketene (I), phosphorylated cyclobutanones (V, VI), and, probably, phosphorylated cyclopropanones (IV), as indicated by the 1810 cm<sup>-1</sup> band. References 4: 3 Russian, 1 Western.  
[207-1386]

UDC 546.185

#### THERMAL DECOMPOSITION OF TETRAALKYLDIAMIDOTRICHLOROPHOSPHORANES

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1417-1418 manuscript received 11 Nov 79

KOVENYA, V. A., MARCHENKO, A. P. and PINCHUK, A. M., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] Tetraalkyldiamidotrichlorophosphoranes were for the first time synthesized in individual state. 0.05 mole of chlorine was passed at 0-5°C through an 0.05 molar solution of tetraalkyldiamidophosphorous acid and 40 cc methylene

chloride. The methylene chloride was then driven off in vacuo and the residue crystallized from a mixture of butyl chloride and ether. The phosphoranes thus synthesized were: tetramethyldiamidotrichlorophosphorane, tetraethylidiamidotrichlorophosphorane, and tetrapropylidiamidotrichlorophosphorane. It was found that on heating these compounds give off alkyl chloride and form phosphazocompounds--with the exception of tetramethyldiamidotrichlorophosphorane, whose thermal decomposition results in the formation of methyl chloride and products of an unidentified structure. References: 2 Russian.

[207-1386]

UDC 547.222:547.241

#### HALOPHILIC INTERACTION OF TRIPHENYLPHOSPHINE WITH HEXACHLOROCYCLOPENTADIENE

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1415-1416 manuscript received

TIMOKHIN, B. V., KRON, V. A. and DUDNIKOVA, V. N., Irkutsk State University imeni A. A. Zhdanov

[Abstract] Reactions between hexachlorocyclopentadiene (I) and tertiary phosphines are so far unknown. It is known, however, that trialkylphosphites react with (I) to form products of the alkylation of (I). It is assumed that these processes commence with the nucleophilic attack by the phosphite against the chlorine atom in the allyl position of the cycle. However, the formation of the corresponding chlorophosphonium cation has not been demonstrated. In this connection, the interaction of (I) with triphenylphosphite (II) was investigated. It turned out that the above reactants readily interact in ether even at temperatures of the order of -80°C. The related NMR and thin-layer chromatography findings indicate that this reaction is accompanied by a halophilic attack by the phosphorus-containing reactant against the chlorine atom in the (I) cycle, with formation of triphenylchlorophosphonium cation. That cation is probably responsible for the conversion of alcohols to chloroalkanes, such as occurs when alcohols are reacted with triphenyldichlorophosphorane, which has the structure of chlorophosphonium salt  $(C_6H_5)_3^{\#}PCl \cdot Cl^-$ . The products of the transformation of the then forming pentachlorocyclopentadienyl anion have not yet been identified. References 3: 2 Russian, 1 Western.

[207-1386]

UDC 547.26'118

#### ALCOHOLYSIS OF ALPHA-PHOSPHORYLATED TRIALKYLPHOSPHITES

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1413-1414 manuscript received 3 Dec 79

KONOVALOVA, I. V., MIRONOV, V. F., OFITSEROVA, Z. Kh. and PUDOVIK, A. N., Kazan' State University imeni V. I. Ul'yanov-Lenin

[Abstract] There exist various techniques for the synthesis of O,O-dialkyl[ $\alpha$ -diethylphosphon)alkyl]phosphites. This is an interesting class of compounds whose chemical properties have not previously been investigated. In this connection, diethyl[ $\alpha$ -(diethylphosphon)benzyl]phosphite (I) was experimentally reacted with ethyl and isopropyl alcohols. This resulted in the formation of diethyl ( $\alpha$ -oxybenzyl)phosphonate and the corresponding trialkylphosphite, with much higher yields than when the standard etherification in the presence of catalysts is employed. It is assumed that such an unusually smooth course of the reetherification reaction is due to the contributing effect of the molecule of the phosphonato-phosphite itself: the formation of a hydrogen bond between the alcohol's proton and the oxygen of the phosphoryl group of the phosphonatophosphite resulting in making the oxygen atom of the alcohol more nucleophilic, which facilitates its subsequent attack against the second fragment of the molecule, which contains the p<sup>III</sup> atom. This assumption is corroborated by the fact that, under analogous conditions, diethyloctylphosphite does not interact at all with ethyl alcohol.

References: 5 Russian.

[207-1386]

UDC 547.455:546.183

#### SYNTHESIS AND TRANSFORMATIONS OF 1-TETRAALKYLDIAMIDOPHOSPHITES OF 2,3;4.5-DIACETONEFRUCTOSE

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 6, 1980 pp 1405-1409  
manuscript received 11 Dec 78

NIFANT'YEV, E. Ye., GUSEYNOV, I. I., ABBASOV, E. M., KOROTEYEV, M. P. and YERMISHKINA, S. A., Moscow State Pedagogical Institute imeni V. I. Lenin

[Abstract] In view of the fact that, compared with aldose chemistry, ketose chemistry is more complicated and relatively uninvestigated, the synthesis and transformations of 1-tetraalkyldiamidophosphites of 2,3; 4.5-diisopropylidene- $\beta$ -D-fuctopyranose were investigated. Phosphorylation of (I) with the aid of hexamethyl- and hexaethyl-triamides of phosphorous acid at 90-100°C for 3-4 hr was found to be an effective technique. Subsequent vacuum distillation resulted in the synthesis of previously undescribed 1-tetralkyldiamidophosphites (II) of (I). Transformations of the resulting phosphamides (II) into compounds with tetra-coordinated phosphorus atom were investigated by (A) adding oxygen and sulfur, and (B) reacting (alkylating) with benzyl bromide. In case A it was found that compounds (II) readily combine with oxygen and sulfur. In case B it was found that, when alkylated with benzyl bromide, 1-diethylamidophosphite of (I) yields 1-bromo-1-desoxydiisopropylidenefructose. References 5: 4 Russian, 1 Western.

[207-1386]

UDC 547.26'118

## AMIDATION OF DERIVATIVES OF S-(ALKOXYCARBONYLMETHYL)DI-, TRI-, AND TETRAPHOSPHORIC ACIDS

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 6, 1980 pp 1325-1329 manuscript received 24 Sep 79

ANDREYEVA, I. A., SOYFER, R. S. and MANDEL'BAUM, Ya. A., All-Union Scientific Research Institute of Chemical Crop Protectants

[Abstract] Derivatives of S-(alkoxycarbonylmethyl)di-, tri-, and tetraphosphoric acids having the general formula  $R'R''P(S)(SCH_2COOR)$  (I), where  $R = Me, Et$ ;  $R'$  and  $R'' = alkoxy, aryloxy-, alkylamide, or alkoxy carbonylmethylthiogroup$ , have been previously synthesized. These derivatives display marked pesticidal activity. In this connection, substitution of the alkoxy carbonylmethyl group with an alkylamino-carbonylmethyl group by means of amidation of compounds (I) can result in synthesizing corresponding derivatives of S-(alkylaminocarbonylmethyl)di-, tri-, and tetraphosphoric acids with a different biological activity. It was shown that interaction of esters of (I) with primary amines yields various amidation products depending on the substituents at the phosphorus atom. The substituted S,S-di(alkoxy-carbonylmethyl)trithiophosphoric acids, when reacted with amines, yield corresponding derivatives of S-(alkoxycarbonylmethyl)-S-(alkylaminocarbonylmethyl)trithiophosphoric acids and diamides of dithioglycolic acid, which form owing to cleavage of the P-S bond. The reaction between S,S,S-tri(alkoxycarbonylmethyl)tetrathio-phosphates and amines results in the phosphorylation of the amine, i.e., in the synthesis of alkylamides of S,S-disubstituted trithiophosphoric acids.

References: 8 Russian.

[207-1386]

UDC 547.26'118

## ALKALINE HYDROLYSIS OF DERIVATIVES OF S-(ALKOXYCARBONYLMETHYL)DI-, TRI- AND TETRATHIOPHOSPHORIC ACIDS

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 6, 1980 pp 1322-1325 manuscript received 13 Aug 79

ANDREYEVA, I. A., MANDEL'BAUM, Ya. A. and SOYFER, R. S., All-Union Scientific Research Institute of Chemical Crop Protectants

[Abstract] In view of the known marked biological activity of derivatives of S-(alkoxycarbonylmethyl)di-, tri-, and tetrathiophosphoric acids, it was of interest to investigate their alkaline hydrolysis, since the amenability of organophosphorus compounds to alkaline hydrolysis is a direct function of their insecticidal activity. The more resistant to hydrolysis that an organophosphorus compound is, the more active cholinesterase inhibitor it is. It was found that the hydrolysis of monoalkylamides of S,S-di(alkoxycarbonylmethyl)-trithiophosphoric acids (Ia) entails the formation of dithioglycolic acid diamide. The hydrolysis of derivatives of (I) entails cleavage of the P-S bond and the formation of

dithiodiglycolate. The hydrolysis of monoalkylamides of S-(alkoxycarbonylmethyl)-di- and trithiophosphoric acids entails cleavage of the P-N bond and the subsequent amidation of the then forming dithiodiglycolate. References 10: 3 Russian, 7 Western.  
[207-1386]

UDC 547.78'558:632.951

#### N'-PHOSPHORYLATED N''-(1-PHENYL-3-METHYLHYDANTOIN-5-YL)UREAS

Leningrad ZHURNAL OБSHCHEY KHIMII in Russian No 6, 1980 pp 1319-1322 manuscript received 6 Aug 79

MYL'NIKOVA, L. M., MANDEL'BAUM, Ya. A. and SOYFER, R. S., All-Union Scientific Research Institute of Crop Protectants

[Abstract] N''-(1,3-disubstituted hydantoin-5-yl)-N'-phosphorylated ureas and thioureas (I) were synthesized with the object of investigating their biological activity. The synthesis of compounds (I) was accomplished by using 1,3-disubstituted 5-amino hydantoins and phosphorus-containing isocyanates in methylene chloride at 20°C for 8-10 hr, with a yield of ~ 90%. Compounds (I) are represented by white crystalline substances that are soluble in acetone, relatively insoluble in benzene and alcohol, insoluble in hydrocarbons and water. Tests of compounds (I) for biological activity demonstrated their marked fungicidal activity--though somewhat inferior to that of the standard fungicide, tetramethylthiuram disulfide. One of compounds (I), namely, N''-(0,0-dialkylthiophosphoryl)-N''-(1-phenyl-3-methylhydantoin-5-yl)urea, when condensed in the presence of oxalylichloride, yields N-(1-phenyl-3-methylhydantoin-5-yl)parabanic acid. References 8: 7 Russian, 1 Western.  
[207-1386]

UDC 546.185

#### ON THE AMINOLYSIS OF 2,2,4-TRICHLORO-1,3-DIAZA-2-PHOSPHORINES

Leningrad ZHURNAL OБSHCHEY KHIMII in Russian No 6, 1980 pp 1313-1319 manuscript received 6 Jul 79

KORNUTA, P. M., KUZ'MENKO, L. S. and KALININ, V. N., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] The substitution order of chlorine atoms in 2,2,4-trichloro-1,3-diaza-2-phosphorines (I) was investigated by studying the aminolysis of (I) with the aid of spectral methods. Dimethyl- and diethylamines and anilines were used as examples. The reaction was performed in benzene solution at 10-20°C, with the above amines being treated with diazaphosphorines (I) having different substituents in positions 5 and 6 of the ring. NQR,  $^{35}\text{Cl}$ , and PMR spectroscopic techniques revealed that in the reaction between 5,6-substituted (I) the first to be exchanged for the amino

group is the chlorine in position 4 of the ring. In addition, decelerated internal rotation of the dimethylamino group about the C<sup>4</sup>-N bond was discovered in dimethylaminoderivatives of 1,3-diaza-2-phosphorines. This appears to be due to the marked weakening of the electron-acceptor properties of the phosphorus atom upon the addition of two-electron-donor dimethylamino groups. References 7: 5 Russian, 2 Western. [207-1386]

UDC 547.241

## ELECTROPHILIC SUBSTITUTION REACTIONS IN THE PHOSPHORYLATED HETEROCYCLIC AMINE SERIES

Leningrad ZHURNAL OBSHCHEY KIMII in Russian No 6, 1980 pp 1309-1313 manuscript received 28 Aug 79

RAZVODOVSKAYA, L. V., GRAPOV, A. F., LI, V. D. and MEL'NIKOV, N. N., All-Union Scientific Research Institute of Chemical Crop Protectants

[Abstract] It is known that, depending on the nature of the heterocycle, the thiophosphorylation of 2-amino-heterocycles results in the formation of substitution products with respect to either one or two atoms of nitrogen or their mixture. In this connection it was of interest to investigate electrophilic substitution reactions in the series of phosphorylated and thiophosphorylated heterocyclic amines containing the amidine system, with the object of elucidating the effect of the nature of the heterocycle and electrophilic reagent on the course of these reactions. The phosphorylated heterocyclic amines used were phosphoryl- and thiophosphorylaminothiazoles, -aminothiazolines, and -aminothiadiazoles. The electrophilic reagents used were methyl iodide, dimethylsulfate, benzenesulfochloride, thiophosphorus acid chlorides, and arylisocyanates. It was found that thiophosphorylated 2-aminothiazoles and 2-aminothiadiazoles react with thiophosphorus acid chlorides, benzenesulfochloride, and arylisocyanate and do not get alkylated by alkyl halides. As for thiophosphorylated 2-aminothiazolines, they react only with arylisocyanates. As for phosphorylated aminoheterocycles, they do not enter into electrophilic substitution reactions. This is due not only to the lower nucleophilicity of the endocyclic nitrogen atom owing to the marked electron-acceptor effect of the phosphoryl group, but also to the presence of a strong intramolecular bond between the phosphoryl oxygen and the proton at the nitrogen atom in position 3. References: 4 Russian.

[207-1386]

UDC 547.241+547.341

INTERACTION OF DIPHENYLCHLOROPHOSPHINE WITH CERTAIN KETONES

Leningrad ZHURNAL OBSCHEY KHIMII in Russian No 6, 1980 pp 1297-1301 manuscript received 13 Jul 79

NURTDINOV, S. Kh., KASHIRSKAYA, I. M., ISMAGILOVA, N. M., GUBAYDULLINA, R. Sh., ZYKOVA, T. V., SALAKHUTDINOV, R. A. and TSIVUNIN, V. S., Kazan' Chemico-Technological Institute imeni S. M. Kirov

[Abstract] Reactions between saturated ketones and primary chlorophosphines are known to yield oxaphospholenes, and reactions between saturated ketones and secondary chlorophosphines are known to yield oxides of tertiary phosphines. In this connection, it was of interest to investigate reactions between diphenylchlorophosphine (I) and acetone or cyclohexanone, as well as between diethylchlorophosphine (II) and certain unsaturated ketones. It was found that, unlike (II), (I) reacts with saturated ketones to form, in the case of acetone, diphenylphosphinic acid and its chloride; and in the case of cyclohexanone, a mixture of phosphorus-containing products. In addition, investigation of the reaction between (II) and acetylacetone points to the possibility of the formation of diethylphosphinous acid during the initial stage of that reaction, as corroborated to some extent by the segregation of dibutylphosphorous acid from the reaction mixture of diethylchlorophosphite and acetylacetone. References 17: 13 Russian, 4 Western. [207-1386]

UDC 542.91:547.379.8:547.341

REACTION OF CERTAIN SELENIUM URANILIDE DERIVATIVES OF DIMEDONE AND 1,3-INDANDIONE WITH TRIPHENYLPHOSPHINE AND TRIS(DIMETHYLAMINO) PHOSPHINE

Leningrad ZHURNAL OBSCHEY KHIMII in Russian No 6, 1980 pp 1294-1296 manuscript received 5 Jul 79

BELKIN, Yu. V., POLEZHAYEVA, N. A., MAGDESIYEVA, N. N. and KYANDZHETSIAN, R. A., Chemical Institute imeni A. M. Butlerov, Kazan' State University imeni V. I. Ul'yanov-Lenin; Moscow State University imeni M. V. Lomonosov

[Abstract] Triphenylphosphine and tris(dimethylamino)phosphine are known to demethylate methylphenylseleniumuranylide derivatives of dimedone and bibenzoylmethane so as to form corresponding phosphonium salts. However, no analogous phosphonium salts have been identified in reactions between dibenzylseleniumuranylide derivatives of dicyanomethane and cyanoacetic ether, on the one hand, and triphenylphosphine, on the other. In this connection, it is now demonstrated that dealkylation of the ilide represents the first stage of its reaction with triphenylphosphine and tris(dimethylamino)phosphine not only in the case of methylphenylseleniumuranylide derivatives but also in the case of their dialkylseleniumuranylide analogues. The reactions between dimethylseleniumuranylide derivatives of dimedone (I) and 1,3-indandione (II), on the one hand, and triphenylphosphine on the other

were carried out in the presence of sodium tetraphenylboranate which served to convert phosphonium salts to the stable form of methyltriphenylphosphonium, thus demonstrating demethylation of ilides (I) and (II) by triphenylphosphine. The reactions between ilides (I) and (II) and tri(dimethylamine)phosphine similarly resulted in the formation of methylhexamethyltriamidephosphonium salts, showing that demethylation took place in this case as well. References 8: 4 Russian, 4 Western.  
[207-1386]

UDC 547.241

#### PHOSPHORYLATION OF IMINOANALOGUES OF ALPHA-HALOCARBONYL COMPOUNDS: I. REACTIONS BETWEEN TRICHLOROACETYLIDOCYANIDES AND TRIALKYLPHOSPHITES

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1288-1294 manuscript received 5 Jun 79

SINITSA, A. D., KRISHTAL', V. S. and KAL'CHENKO, V. I., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] The interaction of alpha-halocarbonyl compounds with nucleophilic derivatives of trivalent phosphorus is useful to the synthesis of physiologically active substances. In this connection, the related reactions with iminoanalogues of  $\alpha$ -halocarbonyl compounds, as exemplified by reactions between trialkylphosphites and N-substituted trichloroacetylidocyanides, are investigated. It is established that N-alkyltrichloroacetylidocyanides and N-dialkoxyphosphoryltrichloroacetylidocyanides react with trialkylphosphites so as to form N-alkyl-N-(2,2-dichloro-1-dialkoxyphosphoryl)vinylamidophosphates and N-trichlorovinyl-N',N'-bis-dialkoxyphosphorylimides. Both the resulting vinylamides and vinylimides are reactable electrophilic compounds that energetically interact with amines. References 16: 13 Russian, 3 Western.  
[207-1386]

UDC 547.26'118+541.68

#### ALKYL ETHYL HALIDES OF ALKOXYALKYLPHOSPHONIC ACIDS

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1273-1288 manuscript received 1 Jun 79

KOZHUSHKO, B. N., PALIYCHUK, Yu. A., BOGEL'FER, L. Ya. and SHOKOL, V. A., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] This is a continuation of an earlier investigation which showed that  $\alpha$ ,  $\alpha$ -dichloroalkyl esters containing electron-acceptor substituents at the  $\alpha$ -carbon atom enter into an Arbuzov reaction with dialkyl-halide phosphites. The interaction of  $\alpha$ ,  $\alpha$ -dichloroalkyl esters, with and without electron-acceptor substituents in the  $\alpha$ -position relative to the halide atoms, on the one hand, and

dialkylchloro- and dialkylfluorophosphites, on the other, is now investigated, and it is shown that such interaction results in the formation of the chlorides and fluorides of the alkyl ethers of  $\alpha$ -alkoxy-  $\alpha$ -carbalkoxy-  $\alpha$ -chloromethylphosphonic acid with chiral atoms of phosphorus and carbon. Their diastereomeric anisochronicity is investigated with the aid of the  $^{1}\text{H}$ ,  $^{19}\text{F}$ , and  $^{31}\text{P}$  NMR method. References 24: 15 Russian, 9 Western.

[207-1386]

UDC 547.341

#### INTERACTION OF UNSATURATED BETA-BROMOPHOSPHONITES WITH NITRILIMINES

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1269-1273 manuscript received 14 Aug 79

PLATONOV, A. Yu., KRUGLOV, A. S., CHISTOKLADOV, V. N. and DOGADINA, A. V., Leningrad Technological Institute of Pulp and Paper Industry; Leningrad Technological Institute imeni Lensovet

[Abstract] The interaction between  $\beta$ -bromoalkenylphosphonites (I) and substituted nitrilimines (II) in 1,3-dipolar addition reactions was investigated. The phosphonites were synthesized by bromophosphinizing acetylene hydrocarbons in the presence of oxygen or UV irradiation. The thus isolated dibromoanhydrides were treated with alcohol in the presence of triethylamine and thus converted to the corresponding ethers of  $\beta$ -bromoalkenylphosphonous acids. It is established that the interaction of (I) and (II) follows the common pattern of reactions of the 1,3-dipolar cyclo-addition of  $\alpha$ ,  $\beta$ -unsaturated compounds of 3-coordinated phosphorus ( $3 + 3 = 6$ , with inclusion of phosphorus atom in the cycle). However, there is one difference: in this case the cyclic ylids of phosphorus obtained as intermediate products are capable of eliminating the bromine anion and forming corresponding quasiphosphonium bromide salts. References: 6 Russian.

[207-1386]

UDC 547.241:541.61

#### EFFECT OF SUBSTITUENTS ON VARIATION IN SPIN-SPIN BONDING CONSTANTS OF H-P AND C-P IN NMR SPECTRA OF THE DERIVATIVES OF 4-COORDINATION PHOSPHORUS

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1262-1269 manuscript received 9 Dec 78

KOMAROV, V. Ya., ZAKHAROV, V. I., BELOV, Yu. V. and IONIN, B. I., Leningrad Technological Institute imeni Lensovet

[Abstract] Variations in the spin-spin bonding constants (SSBC) of H-P and spin-spin interaction constants (SSIC) of C-P of phosphoryl and thiophosphoryl compounds (compounds containing 4-coordination phosphorus) were investigated. It is

established that the pattern of variations in  $^{2}\text{J}_{\text{HP}}$  and  $^{3}\text{J}_{\text{HP}}$  constants in these compounds may be attributed to two mechanisms of the transfer of spin polarization--the overlapping of orbitals (with reversal of polarization) and single-center exchange between s- and p-orbitals (with retention of same polarization). Treatment of the phosphorus with electronegative substituents results in an increase in the absolute magnitude of the geminal and vicinal H-P constants owing to the attendant rehybridization and increase in the s-nature of the phosphorus orbitals in the direction of other atoms. Moreover, comparison of  $^{2}\text{J}_{\text{HP}}$  and  $^{3}\text{J}_{\text{HP}}$  constants in phosphoryl compounds and in the corresponding thiophosphoryl compounds points to the existence of an additional mechanism for the transfer of spin polarization, which adversely affects both the geminal and the vicinal constants. This appears to be due to the participation of 3d-orbitals of phosphoryl compounds in the transmission of spin information--a participation which is lower in the case of thiophosphoryl compound owing to their lower population of 3d-orbitals. Furthermore, analysis of orbital population points to a negative contribution by p-orbitals to the SSBC of  $^{1}\text{J}_{\text{CP}}$ . Figure 1; references 36: 13 Russian, 23 Western.  
[207-1386]

UDC 547.341

#### SYNTHESIS OF DIALKYL ETHERS OF 3-CHLORO-1,3-ALKADIENE-2-PHOSPHONIC ACIDS

Leningrad ZHURNAL OBRASHCHEY KHMII in Russian No 6, 1980 pp 1256-1262  
manuscript received 25 May 79

BREL', V. K., DOGADINA, A. V., ZAKHAROV, V. I., IONIN, B. I. and PETROV, A. A.,  
Leningrad Technological Institute imeni Lensoveta

[Abstract] The derivatives of 1,3-alkadienes with phosphorus-containing groupings are of interest chiefly as monomers for the synthesis of fire-resistant plastics and elastomers. In this connection, it is shown that elementary phosphorus-containing analogues of chloroallene--ethers of 1-chloro-2,3-butadiene-2-phosphonic acid, can be synthesized from dialkylichlorophosphites and 1-chloro-butyne-4-ol. On heating or in the presence of catalysts (such as HCl), these compounds get readily isomerized and form phosphonoprenes (isomeric 1,3-alkadiene-2-phosphonates), which when subjected to radical polymerization, are promising monomers for the synthesis of fire-resistant elastomers. The synthesis of phosphonoprenes through the allene-diene isomerization of ethers of 1-chloro-2,3-butadiene-2-phosphonic acid and its homologues--ethers of 1-chloro-4-methyl-2,3-pentadiene-2-phosphonic acid--is described. Figures 3; references 11: 10 Russian, 1 Western.  
[207-1386]

UDC 547.341

## SYNTHESIS OF DIALKYL ESTERS OF 3-ALKOXY-1,3-ALKADIENE-2-PHOSPHONIC ACIDS

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 6, 1980 pp 1246-1256 manuscript received 5 Jul 79

BREL', V. K., DOGADINA, A. V., IONIN, B. I. and PETROV, A. A., Leningrad Technological Institute imeni Lensoveta

[Abstract] The 1,4-elimination of alcohols on proceeding from 1-alkoxy-2-alkenes, represents a technique for forming the 1,3-alkadiene system of bonds. In this connection, as a way of developing research into the synthesis of highly unsaturated organophosphorus compounds, and particularly of the polymerizable 1,3-alkadiene-2-phosphonates (phosphonoprenes), the above technique was used to synthesize 3-alkoxyphosphonoprenes--dialkyl ethers of 3-alkoxy-1,3-butadiene-2-phosphonic acids. The reactants used were alkoxyallenic phosphonates—dialkyl ethers of 1-alkoxy-2,3-butadiene-2-phosphonic acids. Combining them with alcohols resulted in ethers of 1,3-dialkoxy-2-alkene-2-phosphonates. Subsequent 1,4-elimination of the alcohol resulted in the formation of 3-alkoxyphosphonoprenes. Thus a method for synthesizing a new class of unsaturated organophosphorus compounds could be developed by using readily available derivatives of acetylene glycols on the basis of the elimination of alcohols from alkoxyalkenephosphonates by means of alcoholates or hydrogen chloride. Figures 5; references 7: 6 Russian, 1 Western.

[207-1386]

UDC 541.623+547.419.1

## TETRAAMIDODIPHOSPHINE MONOXIDES AND THE CORRESPONDING ISOMETRIC DIAMIDOPHOSPHOROUS ACID CHLORIDES: PHOSPHOROTROPIC TAUTOMERISM IN THE SERIES OF THESE COMPOUNDS

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 6, 1980 pp 1236-1246 manuscript received 16 Jul 79

POSS, V. L., LUKASHEV, N. V. and LUTSENKO, I. P., Moscow State University imeni M. V. Lomonosov

[Abstract] The decrease in the coordination number of phosphorus upon the isomerization of tetraamidodiphosphine monoxides into acid chlorides is accompanied by the disappearance of the phosphoryl group, and hence elucidation of the factors responsible for the direction of the rearrangement serves to clarify the causes of the stability of phosphoryl-containing compounds in general. Moreover, cases in which the thermodynamic stability of the isomers is commensurate and a tautomeric equilibrium (phototropic tautomerism) is observed, are of special interest. In this connection, methods for the synthesis of various tetraamidodiphosphine monoxides and of the corresponding isomeric acid chlorides are proposed as based on the fact that both these groups of compounds exist in a tautomeric equilibrium and that the share of the acid chloride increases with increase in temperature. The corresponding thermodynamic parameters of the conversion of monoxide to acid chloride are

specified. A new vacuum-distillation technique is proposed for removing amine hydrochloride impurities from amidophosphorous compounds, and thus markedly increasing the yield and thermal stability of these compounds. References 23: 17 Russian, 1 Polish, 5 Western.

[207-1386]

UDC 547.853.7:547.241

#### ANION $\sigma$ -COMPLEXES OF PHOSPHORUS COMPOUNDS

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1230-1236 manuscript received 20 Aug 79

ONYS'KO, P. P., GOLOLOBOV, Yu. G., REMENNIKOV, G. Ya. and CHERKASOV, V. M., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] Reactions between dialkylphosphites and 5-nitropyrimidines were investigated with the object of exploring the comparative ability of aromatic—including heterocyclic—compounds to form phosphorus-containing  $\sigma$ -complexes. Dialkylphosphites were selected because it was thought that, unlike trialkylphosphites, they would prove to be sufficiently strong nucleophiles in the presence of bases to form stable  $\sigma$ -complexes with 5-nitropyrimidines. This assumption was corroborated when it was found that the reaction between dimethylphosphite on 5-nitropyrimidine (Ia, R=H) in the presence of triethylamine resulted in the formation of the  $\sigma$ -complex (IIa, R=H, Alk=Me). On the whole dialkylphosphites form stable phosphorus-containing  $\sigma$ -complexes only with respect to the unsubstituted ring atom of the carbon of 5-nitropyrimidines. It was also found that unlike the  $\text{MeO}^-$  and  $\text{RCOCH}_2^-$  anions, dialkylphosphites do not form  $\sigma$ -complexes with respect to the  $\text{C}^2$  atom of nitropyrimidines. Further, in the reactions between 2,4-dimethoxy-5-nitropyrimidine or 4-methoxy-5-nitropyrimidine and dialkylphosphites the nitro group becomes substituted by the phosphoryl group and the reaction itself involves the intermediate formation of  $\sigma$ -complexes with respect to the  $\text{C}^4$  atom. References 13: 9 Russian, 4 Western.

[207-1386]

UDC 547.241

#### VINYL ESTERS OF THE ACIDS OF PHOSPHORUS. REPORT 19: ALPHA-BROMODITHIOISOBUTYRATES IN PERKOV'S REACTION

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 6, 1980 pp 1226-1230 manuscript received 12 Jun 79

GOLOLOBOV, Yu. G. and DANCHENKO, M. N., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] In view of the increasing interest in substituted vinyl esters of phosphorus acids,  $\alpha$ -bromodithioisobutyrates were selected as the subject of such research, since they are relatively stable, lacking hydrogen atoms when in the  $\alpha$ -

position. It was found that the Perkov vinylation reaction between trialkylphosphites and alkyl ethers of  $\alpha$ -bromodithioisobutyric acid was sufficiently selective, with the principal reaction products being the anticipated thiovinylophosphates. Reactions between the sodium salts of dialkylphosphites and bromodithioisobutyrates were found to result in the formation of  $\alpha$ -alkylmercaptothiovinylophosphates. Reactions between triphenylphosphine and hexaalkyltriamidophosphites on the one hand and bromodithioisobutyrates on the other were found to result in the formation of thiovinyliquasiphosphonium salts. References 15: 9 Russian, 6 Western.

[207-1386]

UDC 547.241:541.67

#### RELATIONSHIP BETWEEN SPECTRAL PARAMETERS AND VALENCE ANGLES IN PHOSPHINES

Leningrad ZHURNAL OBŞHCHEY KHIMII in Russian No 6, 1980 pp 1222-1225 manuscript received 20 Jul 79

BEL'SKIY, V. Ye., ROMANOV, G. V., POZHIDAYEV, V. M. and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] The existence of a relationship between the spectral parameters and valence angles in phosphines was investigated for phosphines with known geometries. It was found that chemical shifts in  $^{31}\text{P}$  NMR spectra, spin-spin interaction constants  $J_{\text{pp}}$ , vertical ionization potentials for the undivided phosphorus pair, and stretching vibration frequencies for the PH bond are functions of valence angle for phosphorus in primary, secondary, and tertiary alkyl- and arylphosphines, including  $\text{PH}_3$ . A linear correlation exists between the spectral parameters considered in the phosphine series. This correlation is absent, however, for phosphines with electronegative substituents, and even more so for compounds of 3-coordination phosphorus with PHal, PO, and PN bonds, owing to polar and resonance effects resulting in an additional redistribution of electron density in the bonding orbitals. References 39: 5 Russian, 34 Western.

[207-1386]

UDC 547.226'118.322

#### ACIDOLYSIS OF THE P-S BOND IN THIODERIVATIVES OF THE ACIDS OF TRIVALENT PHOSPHORUS

Leningrad ZHURNAL OBŞHCHEY KHIMII in Russian No 6, 1980 pp 1217-1221 manuscript received 13 Aug 79

OFTSEROV, Ye. N., SINYASHIN, O. G., IVASYUK, N. V., BATYYEVA, E. S. and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] Reactions between triethyltrithiophosphite (I), diethyl ether of phenyldithiophosphorous acid (II), and ethyl ester of ethylphenylthiophosphinous

acid, on the one hand, and benzoic and acetic acids, on the other, were investigated. It was found that in every case the evolution of the reaction was complex, with the formation of a large number of products, one of which always is the ethyl ester of the corresponding thiocarboxylic acid instead of the theoretically anticipated ester of carboxylic acid, which indicates that the mechanism of the acidolysis of the P-S bond differs from that of its oxygen analogues. Thus, these findings also demonstrate that thioderivatives of trivalent phosphorus react with acids by forming acyclic derivatives of phosphorus and carboxylic acid chlorides. Acidolysis of the P-S bond results in replacement of the thiolic group at the P<sup>III</sup> derivative by a hydroxylic group, along with the formation of thiolic ethers of carboxylic acids and oxygen-containing P<sup>III</sup> acids which, in the presence of thiolic substituents at the phosphorus atom, become unstable and decompose into mercaptans. References 18: 13 Russian, 5 Western.

[207-1386]

UDC 547.241+547.558.1/539.136

### $^{31}\text{P}$ - $^{32}\text{P}$ EXCHANGE IN THE ACID CHLORIDES OF PHOSPHORUS

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 6, 1980 pp 1214-1217 manuscript received 20 Jul 79

KHOZLOV, P. S. and SOKOLOVA, G. D., All-Union Scientific Research Institute of Phytopathology, USSR Ministry of Agriculture

[Abstract]  $^{31}\text{P}$ - $^{32}\text{P}$  isotope exchange between the acid dichlorides of phosphorus and radioactive phosphorus trichloride, phosphorus thiotrichloride, and phosphorus oxychloride was investigated. The acid dichlorides of phosphorus used were new representatives of dichlorophosphines as well as dichlorophosphate, dichlorothiophosphate, dichlorophosphonates, dichlorothiophosphonate, dichlorophosphate, and dichlorothiophosphates. The isotope exchange was experimentally established in mixtures of phosphorus trichloride with dichlorophosphines, dichlorophosphate, and dichlorothiophosphate, as well as in mixtures of phosphorus thiotrichloride with dichlorothiophosphonate and dichlorodithiophosphate. This exchange may be attributed to the realignment of substituents at the phosphorus atom. The absence of isotope exchange in mixtures of dichlorophosphates (phosphonates) with phosphorus oxychloride and of dichlorothiophosphate with phosphorus thiotrichloride is apparently due to the attendant difficulty of formation of an intermediate complex between the reagents. These findings demonstrate that the exchange of phosphorus isotopes in the acid dichlorides of phosphorus is a function of their structure. References 10: 8 Russian, 2 Western.

[207-1386]

UDC 547.26'118

REACTIONS BETWEEN DITHIOACIDS OF PHOSPHORUS AND CHLOROSUBSTITUTED ACETONITRILES

Leningrad ZHURNAL OBRASHCHEY KHIMII in Russian No 1, 1980 pp 24-30 manuscript  
received 18 Dec 78

ZIMIN, M. G., ZABIROV, N. G., SMIRNOV, V. N., CHERKASOV, R. A. and PUDOVIK, A. N.,  
Kazan' State University imeni V. I. Ul'yanov-Lenin

[Abstract] Reactions between diisopropylthiophosphoric and diphenyldithiophosphinic acids, on the one hand, and chloro-, dichloro-, and trichloroacetonitriles were investigated. The reaction between these dithioacids of phosphorus and chloroacetonitrile was found to result in the formation of thiophosphorylated acetonitriles, chlorothioacetamide, and thioacid chlorides of phosphorus. When dichloroacetonitrile is used instead of chloroacetonitrile in the reactions, a series of transformations takes place owing to the substitution of the chlorine atom under the action of the dithiophosphate anion and the formation of imidoylethiophosphates. When trichloroacetonitrile is used, the reactions result in the formation of addition compounds with respect to the CN group--imidoylethiophosphates of phosphorus which react with the second molecule of the dithioacid and stabilize to HCl, bis(thiophosphoryl)disulfides, and dichloroacetonitrile. The interaction of imidoylethiophosphates with HCl results in the formation of trichlorothioacetamide and thioacid chlorides of phosphorus. Moreover, imidoylethiophosphates of phosphorus are capable of rearrangement with the formation of N-thiophosphorylated trichlorothioamides. References 14: 9 Russian, 5 Western.

[139-1386]

UDC 541.67:547.26'118

DIPOLE MOMENTS OF ORGANOPHOSPHORUS COMPOUNDS: XVII. SPATIAL STRUCTURE AND CONFORMATIONAL ASPECTS OF CYCLIC ISOCYANATES OF PHOSPHORUS

Leningrad ZHURNAL OBRASHCHEY KHIMII in Russian No 1, 1980 pp 30-36 manuscript  
received 24 Jan 79

ISHMAYEVA, E. A., OFITSEROV, Ye. N., FASKHUTDINOVA, T. A., ZYABLIKOV, T. A.,  
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[Abstract] The orientation of the isocyanate group in 1,3,2-dioxaphosphorinanes was investigated by methods of PMR spectroscopy and dipole moments. In 2-isocyanato-1,3,2-dioxaphosphorinanes with 3- and 4-coordinated phosphorus atoms the principal type of conformation is the form with gauche orientation of the UEP (undivided electron pair) of the phosphorus atom and the  $N=C=O$  group ( $\sim 80\%$ ). This is because the molecular moment in this case is chiefly determined by the reciprocal orientation of the dipoles of the  $P=X$  bonds and the dioxaphosphorinane ring. This finding is in agreement with the general pattern of conformational behavior of organophosphorus compounds. References 30: 23 Russian, 7 Western.  
[139-1386]

UDC 547.247

DECOMPOSITION OF METHYLETHYLDIISOBUTOXYPHOSPHONIUM IODIDE IN ACETONITRILE AND  
METHYLENE CHLORIDE

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 1, 1980 pp 36-39  
received 26 Dec 78

NESTEROV, L. V. and ALEKSANDROVA, N. A., Institute of Organic and Physical  
Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] The dissociation rate of methylethyldiisobutoxyphosphonium iodide in acetonitrile was investigated as a function of temperature and concentration (at 20, 40, 50, and 60°C and at from 0.11 to 0.04 mol·liter<sup>-1</sup>, respectively) in relation to the conductivity of the solution. It turned out that at 50 and 60°C the reaction is of the second kinetic order, while at 20°C it is of the second order only for concentrations of 0.040 and 0.53 mol·liter<sup>-1</sup>, whereas for concentrations of 0.080 and 0.112 mol·liter<sup>-1</sup> it becomes a first-order kinetic reaction. The fact that the decomposition proceeds in accordance with the first kinetic order at high concentrations and low temperatures and in accordance with the second kinetic order at low concentrations and high temperatures demonstrates the two-stage nature of the decomposition reaction. The decomposition rate of the salt in methylene chloride is one order higher than in the more polar acetonitrile. The first- and second-order reaction rate constants are specified. References 10:  
5 Russian, 5 Western.  
[139-1386]

UDC 547.26'118

REACTIONS IN THE TERNARY SYSTEMS TRIMETHYLPHOSPHITE-SODIUM ION-ORGANIC BASE

Leningrad ZHURNAL OBSCHEY KHMII in Russian No 1, 1980 pp 39-44  
manuscript received 26 Dec 76

NESTEROV, L. V. and SABIROVA, R. A., Institute of Organic and Physical Chemistry  
imeni A.Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] On the basis of an analysis of the literature on reactions between the system  $\text{R}-\ddot{\text{O}}$  and hard electrophilic reagents, as well as on the basis of experiments demonstrating that the isomerization of trimethylphosphite in the presence of sodium salts can be inhibited by adding to the reaction mixture a third reagent--a suitable nucleophilic reagent such as triethylamine, completely new directions of reactions are predicted. This finding is a corollary of Pearson's concept of soft and hard acids and bases, used in the analysis of the reactivity of certain organophosphorus compounds. References: 6 Russian.  
[139-1386]

UDC 547.26'118

BIS(DIALKOXYPHOSPHORYL)IMIDES AND DIALKOXYPHOSPHORYLDIALKOXYTHIOPHOSPHORYLIMIDES  
AND CERTAIN OF THEIR PROPERTIES

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 44-47 manuscript  
received 17 Feb 79

GILYAROV, V. A., MATROSOV, Ye. I., BEL'SKIY, F. I. and KABACHNIK, M. I., Institute  
of Organometallic Compounds, USSR Academy of Sciences

[Abstract] Bis(dialkoxyphosphoryl)imides and dialkoxyphosphoryldialkoxythiophos-  
phorylimides were synthesized through hydrolysis of the corresponding dialkyltri-  
methylsilyl[dialkoxyphosphoryl (or thiophosphoryl)imido]phosphates. Certain of  
their properties were then investigated. Thus, their acidity is generally moderate.  
Their complexing properties, as determined by the potentiometric titration method  
in the ethanol-water system (1:1 by volume) are such that in the presence of  $\text{Ca}^{2+}$   
and  $\text{Co}^{2+}$  ions and the ratio  $M : L = 1$  the titration curves completely coincide  
with the neutralization curve of the imide, which points to total absence of com-  
plexing. In the presence of  $\text{Cu}^{2+}$  ions, however, the neutralization curve shifts  
to a more acid region and an insoluble complex forms toward the end of the titra-  
tion. References 12: 9 Russian, 3 Western.

[139-1386]

UDC 541.454

THEORETICAL INVESTIGATION OF THE ELECTRON EFFECTS OF THE METHYL GROUP IN  
PHOSPHORUS AND SULFUR COMPOUNDS

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 47-52 manuscript  
received 12 Feb 79

ZVEREV, V. V., BAZHANOVA, Z. G. and KITAYEV, Yu. P., Institute of Organic and  
Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of  
Sciences

[Abstract] Upon substitution of hydrogen with the methyl group in saturated com-  
pounds of nitrogen and oxygen, the electron charge of the heteroatom is known to  
decrease with a corresponding decrease in the dipole moment of the molecule, i.e.  
then the methyl group acts as the electron acceptor. It is shown in this connection  
that in  $\text{PH}_3$  and  $\text{H}_2\text{S}$  molecules, by contrast, an analogous substitution results in an  
increase in the electron charge of the heteroatom, i.e. that in this case the  
methyl group acts as an electron donor. This is due to the presence of d-orbitals  
in phosphorus and sulfur ( $\pi(\text{CH}_3)-\text{d}_{\pi\pi}^*$  interaction). Then also the electrostatic  
energy of molecule-proton interaction increases, which magnifies the effect of the  
methyl group on proton affinity in compounds of third-period elements, as compared  
with compounds of second-period elements. References 20: 7 Russian, 13 Western.  
[139-1386]

UDC 547.24

## DERIVATIVES OF PHOSPHINIC ACIDS WITH PHENACYL RADICALS

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 1, 1980 pp 61-64 manuscript  
received 2 Feb 79

MOSKALEVSKAYA, L. S. and FEDORVA, G. K., Institute of Organic Chemistry, UkrSSR  
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[Abstract] It is shown that phenylphenacyl- and diphenacylphosphinic acids display a ketogroup reaction when combined with hydroxylamine hydrochloride in the presence of pyridine. In the former case this reaction results in the synthesis of phenylphenacylphosphinic acid oxime and in the latter, in the cleavage of a C-P bond with the formation of acetophenone oxime and phenacylphosphonic acid oxime. The phenylphenacylphosphonic acid interacts with bromine by the methyl group reaction, with substitution of one or two hydrogen atoms depending on the reagent ratio. When reacted with sodium and methyl iodide, the ethers of phenylphenacyl- and diphenacylphosphinic acids undergo methylation with respect to the  $\alpha$ -carbon atom.

References: 3 Russian.

[139-1386]

UDC 547.26'118

## PHOSPHORYLATED BENZIMIDAZOLES. IV: PHOSPHONETHYLATION OF BENZIMIDAZOLE AND ITS DERIVATIVES

Leningrad ZHURNAL OBSHCHEY KHMII in Russian No 1, 1980 pp 64-67 manuscript  
received 24 Jan 79

MATEVOSYAN, G. L., MATYUSHICHEVA, R. M., RUDNIK, S. P. and ZAVLIN, P. M.,  
Leningrad Agricultural Institute; Leningrad Institute of Motion Picture Engineers

[Abstract] As part of the search for effective plant growth regulators, the possibility of synthesizing heterocyclic derivatives of ethylphosphonic acid was explored. To this end, the interaction between benzimidazole and its 5(6)-derivatives and di( $\beta$ -chloroethyl ester)vinylphosphonic acid was investigated. It was found that the attendant phosphonethylation reaction is accomplished with a yield of more than 90% in the presence of Lewis acids, e.g. anhydrous AlCl<sub>3</sub>. It is worth noting that in the presence of KOH such relatively nonbasic derivatives of benzimidazole as 5(6) nitrobenzimidazole also participate in the phosphonethylation reaction. Thus it is possible to perform selective phosphonalkylation of benzimidazole and its 5(6)-derivatives by means of di( $\beta$ -chloroethyl) vinylphosphonate, with the formation of both derivatives of  $\beta$ -(1-benzimidazole)ethylphosphonic acid and di[ $\beta$ -(1-benzimidazolyl)ethyl ester]vinylphosphonic acid. The compounds thus synthesized proved to be effective regulators of plant growth.

References: 3 Russian.

[139-1386]

UDC 547.341

## REDOX POTENTIALS OF CYCLIC DITHIO- AND MONOTHIOPHOSPHATES

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 67-69 manuscript  
received 5 Feb 79

OVCHINNIKOV, V. V., GARIFZYANOV, A. R., CHERKASOV, R. A. and TOROPOVA, V. F.,  
Kazan' State University imeni V. I. Ul'yanov-Lenin

[Abstract] The redox potentials of systems of acyclic and cyclic derivatives of di- and monothioacids of phosphorus were determined in aqueous propyl alcohol and tabulated. For cyclic dithiophosphates these potentials proved to be much higher than for their acyclic analogues, which may indicate lower reducing properties of dithiophosphate groups with cyclic dioxalkylene substituents, which are of a marked electron-acceptor nature. For cyclic monothiophosphates the recorded redox potentials are even more positive than those of the analogous dithiophosphates. A correlation is found to exist between redox potential, the electron effect of the substituents at the phosphorus atom, the stability constants of nickel complexes of the dithioacids of phosphorus, and the reaction rate constants of these acids. References 9: 7 Russian, 2 Western.

[139-1386]

UDC 547.118

## INTERACTION BETWEEN TRIETHYLTRITHIOPHOSPHITE AND ACIDS

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 222-223 manuscript  
received 5 Jun 79

OFITSEROV, Ye. N., SINYASHIN, O. G., BATYEVA, E. S. and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni Ye. A. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] Trialkyltrithiophosphites are known to be inert with respect of acids, and triaryltrithiophosphite is known to yield phosphorous acid when heated in concentrated HCl. By contrast with these data, it is now established that triethyltrithiophosphite reacts with HCl to form diethyldithiophosphorous acid chloride and ethylthiophosphorous acid dichloride. When reacted with benzoic acid (at 120-140°C and up to 100 mm Hg), triethyltrithiophosphite forms ethylmercaptan, ethyl ether of thiobenzoic acid (upon distillation of residue after removal of mercaptan at 0.07 mm Hg), and an incomplete phosphorous anhydride. References 7: 5 Russian, 2 Western.  
[139-1386]

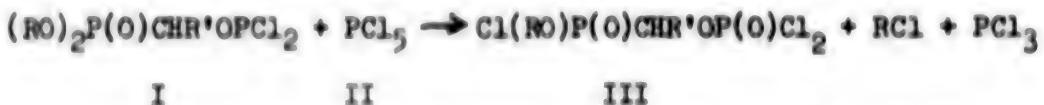
UDC 547.26'118+546.185'181

INTERACTION BETWEEN BISPHOSPHORIC ORGANIC COMPOUNDS WITH VARYING PHOSPHORUS ATOM COORDINATION AND PHOSPHORUS PENTACHLORIDE

Leningrad ZHURNAL OBSCHEY KHIMII in Russian No 1, 1980 pp 223-224 manuscript received 7 May 79

GAZIZOV, M. B., KHAYRULLIN, R. A. and RAZUMOV, A. I., Kazan' Chemico-Technological Institute imeni S. M. Kirov

[Abstract] A new interesting reaction of interaction between bisphosphoric organic compounds with varying phosphorus atom coordination (I) and phosphorus pentachloride (II) is discovered. The reaction is characterized by the simultaneous substitution of the alkoxygroup by chlorine in 4-coordinated phosphorus and the increase in coordination number of the second phosphorus atom. The reaction products are acid chlorides (III), phosphorus trichloride, and alkyl halide.



Compounds I and II react in a 1:1 ratio in benzene solution at 60°C.  
[139-1386]

UDC 547.241+547.73

PHOSPHORYLATION OF 2(DIETHOXYMETHYL)THIOPHENE

Leningrad ZHURNAL OBSCHEY KHIMII in Russian No 1, 1980 pp 224-225 manuscript received 1 Jun 79

NEVZOROVA, O. L., KRASIL'NIKOVA, Ye. A., RAZUMOV, A. I. and KUZNETSOVA, S. A., Kazan' Chemico-Technological Institute imeni S. M. Kirov

[Abstract] The interaction between 2-(diethoxymethyl)thiophene and butyllithium and dialkylchlorophosphites was investigated with the object of developing a convenient technique for the phosphorylation of thiophene and its derivatives. The reaction resulted in the synthesis of dialkyl-2(5-diethoxymethylthienyl)phosphonites which, when reacted with alkyl bromides and acetic aldehyde bromacetal, form alkyl-2-(5-diethoxymethylthienyl)phosphinates and alkyl( $\alpha$ , $\beta$ -diethoxyethyl)-2-(5-diethoxymethylthienyl)phosphinate. The structure of all the synthesized compounds was corroborated by their IR and NMR  $^{31}P$  and  $^1H$  spectra. References: 3 Russian.  
[139-1386]

UDC 547.558.1

UNEXPECTED REACTIONS OF PHENYLTETRAIODOPHOSPHORANE

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 225-226 manuscript received 5 May 79

FESHCHENKO, N. G., KUDRYAVTSEVA, L. I. and KIRSANOV, A. V., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] It is found that, when reacted with sodium phenolate in the ratio 1:4, phenyltetraiodophosphorane ( $C_6H_5PI_4$ ) yields phenyltetraphenoxyphosphorane—a crystalline substance with a high m. p. that is quite readily hydrolyzed by the moisture of the air. Moreover, phenyltetraiodophosphorane reacts with methyl iodide to form a methylation product having the composition  $CH_3(C_6H_5)_3I_2P$ , which is represented by a violet-brown substance with a high m.p. that is readily hydrolyzed and is insoluble in most organic compounds. Following its hydrolysis this substance changes into methylphenylphosphinic acid. References 3: 1 Russian, 2 Western.  
[139-1386]

UDC 547.26'118

THERMAL REARRANGEMENT OF BIS(N-ALKYL-N-PHENYLAMIDO)-CHLOROPHOSPHAZOBENZENES

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 226-227 manuscript received 27 Apr 79

MARCHENKO, A. P., KOVENYA, V. A. and PINCHUK, A. M., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] It is found that bis(N-alkyl-N-phenylamido)chlorophosphazobenzenes undergo thermal rearrangement; this entails elimination of alkyl chloride and the formation of previously unknown derivatives of 1,3,2,4-diazadiphosphetidinediimides. The reaction is performed at 250-270°C in vacuo (60-70 mm Hg) for 2-3 hr in an argon atmosphere. The completion of the rearrangement is determined as a function of the amount of the isolated alkyl chloride. The residue is crystallized. References 2: 1 Russian, 1 Western.  
[139-1386]

UDC 547.26'118

## A NEW METHOD FOR THE SYNTHESIS OF DIALKYLPHOSPHOROUS ACID CHLORIDES

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 227-228 manuscript received 28 May 79

KABACHNIK, M. M., SNYATKOVA, Ye. V., NOVIKOVA, Z. S. and LUTSENKO, I. F., Moscow State University imeni M. V. Lomonosov

[Abstract] The traditional methods for synthesizing dialkylphosphorous acid chlorides are chiefly based on reacting the halide derivatives of that acid with alcohol in the presence of organic bases. Now a new method for the synthesis of dialkylphosphorous acid monochlorides is proposed, consisting in the interaction between equimolar quantities of dialkylphosphites and phosphorus pentachloride or thionyl chloride in the presence of triethylamine. Yield: 42-92% (the higher yields are observed in the case of secondary radicals in the alkylphosphites). It is worth noting that the reaction of dialkylphosphite with thionyl chloride has resulted in the isolation and identification of such byproducts as the corresponding dialkylchlorophosphates and dialkylchlorothiophosphates. The dialkylphosphorous acid chlorides thus synthesized are: diethylphosphorous acid chloride,  $(\text{PrO})_2\text{PCl}$ ,  $(\text{BuO})_2\text{PCl}$ ,  $(i\text{-PrO})_2\text{PCl}$ , and  $(s\text{-BuO})\text{PCl}$ . References 4: 2 Russian, 2 Western.  
[139-1386]

UDC 547.241

## SYNTHESIS OF DIPHOSPHOROUS<sup>V</sup>-SUBSTITUTED PHOSPHINES

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 228-230 manuscript received 28 May 79

KABACHNIK, M. M., PRISHCHENKO, A. A., SNYATKOVA, Ye. V., NOVIKOVA, Z. S. and LUTSENKO, I. F., Moscow State University imeni M. V. Lomonosov

[Abstract] It is shown that alkylidiodophosphines readily react with the acid esters of 3-coordination phosphorus to form diphosphorus<sup>V</sup>-substituted phosphines. The reaction occurs rapidly at -78°C in methylene chloride. The yields of the diphosphorus<sup>V</sup>-substituted phosphines amount to 75-92%. The reaction between alkylidiodophosphines and tetraalkylmethyldiphosphonites occur analogously, and results in the formation of triphosphacyclobutanes. References: 3 Russian.  
[139-1386]

**PHOSPHINES**

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 230-231 manuscript received 26 Apr 79

KOLODYAZHNYY, O. I., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] Recently it has been shown that chlorophosphines ( $I, X=Y=CO_2Et$ ) get readily dehydrochlorinated by triethylamine. However, then then forming phosphenes became dimerized and could not be isolated. On continuing this investigation it is now found that the introduction of the tert-butyl group onto the phosphorus atom results in the stabilization of the phosphenes and their individual synthesis. The thus synthesized phosphenes are: dimethoxycarbonylmethanephosphene and methoxycarbonylphenylimethane-tert.-butylphosphene. The structure of the synthesized compounds was corroborated by their IR and PMR spectra. References 4: 2 Russian, 2 Western. [139-1386]

**SYNTHESIS OF ACID ESTERS OF 3-COORDINATION PHOSPHORUS FROM ACETAL**

Leningrad ZHURNAL OBSHCHEY KHIMII in Russian No 1, 1980 pp 231-232 manuscript received

GAZIZOV, M. B., RAZUMOV, A. I. and KHAYRULLIN, R. A.

[Abstract] It is shown for the first time that the reaction between ethyl- $\beta,\beta,\beta$ -trichloroethyl acetal of acetic aldehyde and  $PCl_3$  in benzene at  $60^\circ C$  in a ratio of 1:3 stops in the stage of the formation of tris( $\beta,\beta,\beta$ -trichloroethyl) phosphite. Similarly, the reaction between that acetal compound and phenyldichlorophosphine stops in the stage of formation of phosphonite. It is assumed that this cessation of reaction is due to the marked decrease in the n-donor properties of the 3-coordinated phosphorus in these compounds under the action of electron-acceptor  $\beta,\beta,\beta$ -trichloroethyl groups. The structure and composition of these compounds are corroborated by their IR and NMR $^1H$  and  $^{31}P$  spectra and by elementary analysis for P, C, and H. Reference: 1 Russian. [139-1386]

UDC 547.26'118

REACTION BETWEEN PHENYL ESTERS OF THE ACIDS OF TRIVALENT PHOSPHORUS AND CARBOXYLIC ACID CHLORIDES

Leningrad ZHURNAL OBŠHCHEY KHMII in Russian No 1, 1980 pp 232-233 manuscript received 5 Jun 79

GAZIZOV, T. Kh., BELYALOV, R. U., KHARLAMOV, V. A. and PUDOVIK, A. N., Institute of Organic and Physical Chemistry imeni A. Ye. Arbuzov, Kazan' Branch of the USSR Academy of Sciences

[Abstract] It is assumed that triphenylphosphite reacts with carboxylic acid chlorides to form diphenylchlorophosphite and the phenyl ester of the corresponding acid on the basis of a preliminary decomposition of the carboxylic acid chloride into ketene and HCl. This assumption was verified by investigating reactions between the phenyl esters of the acids of PIII and the carboxylic acid chlorides which cannot decompose into ketene and HCl. It was found that phenyldibutylphosphinite reacts exothermically with benzoyl chloride at room temperature to form an intermediate product identified as a quasiphosphonium compound, and ultimately to form dibutylchlorophosphine. A similar picture was observed for the reaction of triphenylphosphite with acetyl chloride. Thus these reactions occur with the formation of a quasiphosphonium compound as an intermediate product. Apparently that intermediate product may exist in an equilibrium between ion and covalent forms, and its phenoxyphosphonium form is responsible for the formation of the final reaction products. References 4: 2 Russian, 2 Western.

[139-1386]

UDC 547.241

REACTION BETWEEN TERT.-BUTYL-BIS(TRIMETHYLSILYL)PHOSPHINE AND CARBOXYLIC ACID CHLORIDES

Leningrad ZHURNAL OBŠHCHEY KHMII in Russian No 1, 1980 pp 233-234 manuscript received 25 May 79

KOLODYAZHNYY, O. I. and KUKHAR', V. P., Institute of Organic Chemistry, UkrSSR Academy of Sciences

[Abstract] It was of interest to elucidate the effect of R' substituents associated with the carbon atom of the P=C bond on the properties of phosphenes (compounds of 2-coordination phosphorus containing the P=C bond in an open chain). To this end, the reaction between tert.-butyl-bis(trimethylsilyl)phosphine and the chlorides of various carboxylic acids was investigated. It was found that, depending on the nature of the R' substituent, the final reaction products are either a 3-coordination phosphorus compound, acylsilylphosphine ( $R' = OEt$ ), or compounds of 2-coordination phosphorus, phosphenes ( $R' = Alk, CHPh_2, CO_2Et, CF_3$ ). On the whole the most stable phosphenes are those whose R' substituents display high values of steric constants  $E_s^0$  ( $CHPh_2$ —2.42, t-Bu—2.14,  $CF_3$ —2.15). References 2: 1 Russian, 1 Western.

[139-1386]

## PESTICIDES

### NEW AGENTS FOR THE CONTROL OF PESTS AND WEEDS ON COTTON FIELDS

Dushanbe SEL'SKOYE KHOZYAYSTVO TADZHIKISTANA in Russian No 2, 1980 pp 49-50

[Article by G. V. Dutsof, V. V. Bochkarev and S. N. Pavlov, Tajik Toxicological Laboratory of the All-Union Institute for the Protection of Plants]

[Text] The Tajik Toxicological Laboratory of the All-Union Institute for Protection of Plants has tested more than 150 products, of which the most effective pesticides were selected, which have highly selective action and are minimally toxic to man, animals and useful organisms. Most of them are manufactured by our industry, while some will be produced in 1980. Proper use thereof provides reliable protection of cotton plants against red spider mites (*Tetranychus urticae* Koch) and bollworms.

#### Description of Pesticides

PP-199: 40% colloid solution; poorly soluble in water; has acaricidal, insecticidal and fungicidal properties. It can be used against aphids, mites, scale and whiteflies.

Doses of 0.125 and 0.200 kg/ha [hectare] reduce the number of aphids by 98% on the 5th day after treatment, 97% on the 10th and 91.1% on the 15th day. The spider mite population decreases by 100, 99.4 and 99% after 5, 10 and 15 days, respectively. In 1978, no spider mites were found on treated fields 5, 10 and 15 days after using this agent.

Kroneton: 50% emulsifying concentrate. It is a chemically pure substance consisting of clear [colorless] crystals. The commercial product is a yellowish oil. It is a systemic insecticide for the control of aphids. It is effective against aphid populations that are resistant to organophosphorus products. It is moderately toxic for warm-blooded animals.

A dosage of 1 kg/ha lowers the number of aphids by 92.3 and 99.3% 5 and 10 days, respectively, after treatment. It has no adverse effect on plants.

Sumicid: 20% emulsifying concentrate. The compound is stable in organic solvents, with the exception of alcohol. It is a contact, enteral insecticide with a new mechanism of action, differing in this respect from chlorophosphorocarbamate derivatives. Its insecticidal action is rapidly manifested with the use of low doses against a wide range of pests of different plants.

A dosage of 0.5 kg/ha destroys all aphids 50 and 10 days after treatment and reduces the number of bollworms by 96.9-100%.

Pirimor: 50% wetting powder; a selective aphicidal agent with rapid action. It is effective in the control of various types of aphids, including those that are resistant to organophosphorus insecticides. Use of 2 kg/ha reduces the number of aphids by 86 and 93% 5 and 10 days, respectively, after treatment. This agent penetrates into leaves; it has deep, contact and fumigant action. Pirimor is hazardous to useful insects. Appropriate precautionary measures must be taken when working with it.

Isofen: 50% wetting powder of domestic manufacture. It is toxic to phytophagous mites at all stages of development. It is effective against mites that are resistant to organochlorine and organophosphorus acaricides. A dosage of 3 kg/ha reduces the number of spider mites by 98, 95 and 96% 5, 10 and 15 days, respectively, after treatment.

Tork: 50% wetting powder. A contact-action acaricide, which is effective against mites at all stages of development, including those resistant to organochlorine and organophosphorus compounds. When used in a dosage of 2.5 kg/ha, it reduces the number of spider mites by 95 and 98% 5 and 10 days after treatment; in 1978, invasiveness decreased by 89, 92 and 91% 5, 10 and 15 days, respectively, after use of this agent. It has no adverse effect on cotton plants.

Kayakhop: 50% wetting powder. It is a contact acaricide against phytophagous mites at all stages of development which have ovicidal activity. A dose of 2 kg/ha reduces the number of spider mites by 89, 98 and 97% 5, 10 and 15 days, respectively, after treatment; in 1978, the figures were 86, 90 and 88%, respectively.

Mugan': 85% wetting powder of domestic manufacture. It is used against pests, for the control of which sevin had been used previously, and in particular against the bollworm. It reduces the number of bollworm caterpillars by 88 and 100% with the use of 2.5 kg/ha of the product and by 96 and 100% with the use of 3 kg/ha 5 and 10 days after treatment.

Detsis: 2.5% emulsifying concentrate. Its active ingredient is synthetic pyrethrin (ester of cyclopropanic acid consisting of one active

isomer). It is a contact-enteral insecticide with acaricidal properties. In a dosage of 1 kg/ha, it reduces the number of bollworms by 92 and 93% 5 and 10 days after treatment.

Dikofol: 40% wetting powder of domestic manufacture. The active ingredient contains 18% 4,4-isomer. This acaricide is effective against resistant mites. A dosage of 4 kg/ha reduces the number of spider mites by 99% 5 days after treatment and by 98-94% on the 10th-15th days. It has no adverse effect on cotton plants.

Pliktran (standard): 25% wetting powder. The active ingredient is stannic tricyclohexyl hydroxide. It is a contact acaricide, toxic to adult mites and larvae. It has repellent properties against some phytophagous insects. A dosage of 3 kg/ha reduces the number of spider mites by 99, 99.7 and 98% 5, 10 and 15 days after treatment. It has no adverse effect on cotton plants.

#### Description of Herbicides

Toluin: 30% wetting powder. Its active ingredient is methoxyethyl chloracetotoluidide. It is used against annual weeds before cotton plants sprout. Dose of 10 and 16.7 kg/ha reduce the amount of weeds on cotton fields by 84 and 92%. It has no adverse effect on cotton plants.

Pakhtoron: 80% wetting powder. It is used to control annual weeds, both at sowing time and before cotton plants sprout. A dosage of 2 kg/ha reduces the amount of weeds by 73-84%. It has no adverse effect on cotton plants.

[156-10,657]

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10,657  
CSO: 1841

UDC 547.364

INSECT PHEROMONES AND THEIR ANALOGS: III. SYNTHESIS OF SEX ATTRACTANTS OF SOME LEPIDOPTERA

Tashkent KHIMIYA PRIRODNYKH SOYEDINENIY in Russian No 1, 1980 pp 97-102 manuscript received 14 Jul 79

DEHEMILEV, U. M., BALEZINA, G. G., VOLKOVA, L. A., KRIVONOGOV, V. P. and TOLSTIKOV, G. A., USSR Academy of Sciences Bashkir Branch Institute of Chemistry, Ufa

[Abstract] A new approach is suggested for synthesizing a number of Lepidoptera attractants by using as starting compounds accessible unsaturated esters of butadiene with methyl acrylate. Specifically, a new method is presented for synthesizing a number of attractants of *Argyrotaenia velutinana*, *Mamestra configurata* and *Lycorea ceres ceres* on the basis of combining C<sub>3</sub>-, C<sub>5</sub>- and C<sub>7</sub>-aldehydes with the methyl ester of 11-bromoundecyclic acid according to the Wittig reaction. Proprio-, n-valer- and n-heptaldehydes were used as C<sub>3</sub>-C<sub>7</sub> fragments. These were synthesized from a mixture of 2Z(E)-5E-heptadienic acids (I) by the gradual hydrogenation of I on skeleton nickel, the reduction of LiAlH<sub>4</sub> in ether and the oxidation to the heptyl aldehyde (II) according to the Corey method (1975). For the purpose of producing the methyl ester of 11-bromoundecyclic acid esters of 2E and 5E and 10-undecatrienic (III) and 10-undecylenic (IV) acid were used. The hydroboration of III with B<sub>2</sub>H<sub>6</sub> in THF followed by treatment of the reaction products with 30 percent H<sub>2</sub>O<sub>2</sub> in an alkaline medium produces a mixture of unsaturated alcohols V and VI in a ratio of 9:1 with a total yield of 50 percent in terms of the original ether. The hydrogenation of V and VI on skeleton nickel in ethyl acetate results in a mixture of methyl ethers of 11-(10)-oxyundecyclic acids VII and VIII with a quantitative yield. Treatment of the alcohol of VII with PBr<sub>3</sub> in benzene produces an 11-bromoundecanoate (IX) with a yield of about 85 percent. By reacting the triphenyl phosphonic salt of IX with the aldehydes mentioned in a medium of dimethyl formamide under the effect of tert-BuOK methyl esters of 11Z-tetradecene-carbonic (X), 11Z-hexadecene-carbonic (XI) and 11Z-octadecenecarbonic (XII) acids are produced with yields of 65 percent. The reduction of X to XII by the means of LiAlH<sub>4</sub> in ether results in alcohols XIII to XV, the acetylation of which with (CH<sub>3</sub>CO)<sub>2</sub>O in pyridine results in quantitative yields of attractants of *Argyrotaenia velutinana* (XVI), *Mamestra configurata* (XVII) and *Lycorea ceres ceres* (XVIII). The procedures for producing all of these substances are described in detail. References 13: 6 Russian, 7 Western. [145-8831]

UDC 633.88.632.954

HERBICIDES IN LEUZA CARTHAMOIDES D.C. SOWINGS

Moscow KHLIMIKO-FARMATSEVTICHESKIY ZHURNAL in Russian No 4, 1980 submitted  
24 Apr 79 signed to press 27 Mar 80 pp 55-57

VEKSHIN, B. S., BUKINA, N. V., PUSHKINA, G. P. and YEFIMOVA, V. N., All-Union  
Scientific Research Institute of Medicinal Plants, Moscow Oblast

[Abstract] The resumption of tests of herbicides on leuza in 1975 indicated that patoran and arezin provided the best weed control and degree of selectivity on crops in the first year of vegetation, while dual, azulox, treflan and karakhol were adequate but kotoran was highly toxic. Tests on plantations in the second and third year of growth showed linuron and patoran to be most effective in this period, keeping the plantings weed-free throughout the vegetation period and providing root yields equal to control plantings.

[183-2791]

UDC 543.544

SYSTEMS OF GAS-CHROMATOGRAPHIC RETENTION INDICES FOR IDENTIFICATION OF PESTICIDES  
USING SELECTIVE DETECTORS

Moscow ZHURNAL ANALITICHESKOY KHMII in Russian Vol 25, No 5, 1980 pp 964-968  
manuscript received 21 May 79

GOLOVKIN, G. V., SMOL'CHENKO, A. I., ZOTOV, L. N., SAZONOV, S. Ye., All-Union  
Scientific Research Institute of Plant Protection Chemicals, Moscow

[Abstract] The retention indices for a number of compounds are presented on specific specific scales convenient for qualitative analysis of pesticide residues in natural objects. The investigation was carried out on the Tsvet-110 chromatograph with thermionic, electron-capture and flame-ionization detectors and on the model 419 Packard chromatograph (United States) with flame-photometric detector. A series of trichloroacetic acid esters were used for the electron-capture detector, trialkyl-phosphates were used for the thermionic detector and a series of symmetric dialkylsulphides were used for the flame-photometric detector. The halogen, phosphorus and sulphur retention indices are recalculated to Kovats retention indices and vice versa in the new system of indices convenient for gas-chromatographic analysis of pesticides using selective detectors. Three polysiloxane stationary phases SE-30, OV-17 and XE-60 were used for reliable identification of a large number of pesticides in environmental samples. References 9: 6 Russian, 3 Western.  
[189-6521]

UDC 547.898

CROWN-ETHER CHEMISTRY IX SYNTHESIS OF MACROCYCLES CONTAINING DOUBLE C=C BONDS AND STUDY OF THEIR BIOLOGICAL PROPERTIES

Yerevan ARMYANSKIY KHMICHESKIY ZHURNAL in Russian No 4, 1980 submitted 6 Dec 79 signed to press 30 Apr 80 pp 308-310

VARTANYAN, S. A., AKOPYAN, T. R., PARONIKY, Ye. G., and DARBINYAN, G. A., Armenian SSR Academy of Sciences Institute of Organic Chemistry imeni A. L. Midzhoyan, Yerevan

[Abstract] New macrocycles containing double bonds were produced by the interaction of dioxybenzenes and tarsa-1,4-dichloro-2-butene and tetrabromoderivatives of them were produced by bromination. A study of the mutagenic effect of the synthesized compounds with respect to auxotrophic strains of *E. coli* P-678 thr and *Actinomyces rimosus* 222 lys<sup>-</sup> showed that 3 of the 6 produce a moderate mutagenic effect at the threonine locus of coliform bacterium, inducing mutations 100 to 226-fold greater than the control (spontaneous mutation) and 675-980 times greater than the control in respect to the lysine locus of actiomycetes. References 2; Figure 1.

[213-2791]

PHARMACOLOGY AND TOXICOLOGY

UDC: 577.152:577.15.047

STUDY OF THE EFFECTS OF AMINOESTERS OF THE CYCLOHEXENE SERIES ON CHOLINESTERASE AND TRYPSIN ACTIVITY

Minsk DOKLADY AKADEMII NAUK BSSR in Russian No 4, 1980 pp 337-340

[Article by N. S. Kozlov, academician of the Belorussian Academy of Sciences, I. G. Sheherbak, V. I. Cherepok, L. V. Gladkikh, A. I. Kulikova and F. A. Tugusheva, Institute of Physical and Organic Chemistry, Belorussian Academy of Sciences, and First Leningrad Medical Institute imeni Academician I. P. Pavlov, submitted 21 May 79]

[Text] Many aminoesters of the aromatic and aliphatic series have found broad use as drugs, the effects of which are related, in a number of cases, to the anticholinesterase properties of the compounds [1]. In order to broaden the arsenal of such agents and find more effective ones, it was interesting to investigate the effects of aminoesters of the cyclohexene series on activity of acetylcholinesterase (ACE), butyryl cholinesterase (BuCE) and trypsin. The choice of expressly these enzymes, which contain the amino acid serine in the structure of their active center, is attributable to their extensive presence in animal tissues and involvement in implementation of diverse physiological functions.

The Table lists data on the results of testing 11 compounds of the cyclohexene series, which are water-miscible salts of amino ethers or esters.

Compounds 1-6, 8 and 9, which contain an ester group, could theoretically be the substrates of the tested enzymes. Nevertheless, these esters were not hydrolyzed by either trypsin or cholinesterases. The resistance of the compounds to hydrolysis by trypsin is attributable to the absence of residues of the main amino acids, arginine or lysine, in the acyl part of the molecule, while the inability of cholinesterases to split these substances is apparently related to the reverse orientation of the ester group, as compared to acetylcholine, which is the natural substrate of these enzymes.

Compounds with an ester group have different effects on trypsin activity. Compounds 1, 3, 5, 6, 8 and 9 inhibit trypsin activity by 50-70%, whereas

compounds 2, 4 and 5 do not have this property. Consequently, appreciable antitrypsin properties are demonstrable in the compounds studied in the cases when the atom of nitrogen is a part of the dimethylamino group (1), trimethylammonia (3) or the morpholine ring (6, 8, 9). A comparison of the activity of compounds 1, 3, 4 and 5, 6, 8 shows that quaternization of nitrogen does not affect the degree of antitrypsin properties of the compounds studied. The absence of antitrypsin properties in cyclohexene aminoesters is related to the presence of nitrogen in the piperidine ring (4, 5) or diethylamino group (2).

The anticholinesterase properties of cyclohexene esters are the most marked in salts of tetrasubstituted ammonia with dimethylamine and piperidine groups (compounds 3 and 5). It is known [2, 6-8] that the presence of a charge on the atom of nitrogen facilitates interaction between a compound and active surface of anion center cholinesterases. The nitrogen-containing part of compound 3 is closest in structure to the cation head of acetylcholine; for this reason, this compound is the most potent anticholinesterase agent of all those listed in the table. Hydrophobic interaction of nonpolar groups of substrate or inhibitor with analogous segments of the molecule of enzyme surrounding the anion or esterase center of cholinesterases is equally important to formation of substrate-enzyme or inhibitor-enzyme complexes. The more hydrophobic piperidine ring satisfies this condition, while the morpholine structure most probably does not. The significant capacity of compound 8 to inhibit BuCE can be attributed to the marked hydrophobia of the aromatic radical bound with the morpholine structure and spatial conformity of this radical with the structure of the hydrophobic environment of the anion center which, as we know [2], is considerably more marked in BuCE than in ACE.

Unlike the above-listed compounds, agents 7, 10 and 11 contain a simple ether bond. The table shows that amino ethers ["simple amino esters"] with a primary amino group [7, 10] do not have significant activity with regard to the tested enzymes, although we know from the literature [3] that n-butylamine and benzylamine have appreciable antitrypsin properties. Hence, it may be concluded that a cyclohexene structure combined with an ether bond prevents manifestation of inhibitory properties for trypsin. However, compound 11 has appreciable antienzyme properties, but they are apparently related to the presence of a trioxyethylammonia group, in which the atom of nitrogen is connected, like in the morpholine ring, to oxygen through two methylene bridges.

The obtained results warrant the conclusion that the presence of a piperidine ring (compounds 4 and 5), particularly combined with tetrasubstituted atom of nitrogen, serves as an obstacle to expression of antitrypsin activity and, at the same time, enhances interaction with cholinesterases. The presence of a morpholine ring in cyclohexene compounds, conversely, imparts appreciable antitrypsin properties to these compounds, against the background of absent or minimally marked anticholinesterase properties. Quaternization of the atom of nitrogen is not significant to expression of antitrypsin activity, unlike anticholinesterase action.

Degree of depression (%) of purified ACE, BuCE and trypsin preparations in the presence of different concentrations (M) of cyclohexene aminoesters

No	Formula of compound	Trypsin		ACE		BuCE	
		$\frac{1}{2}$	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{1}{4}$
1		55±3	23±6			40±2	
2		8±3	18±6			7±3	
3		50±7	98±2	44±10	4±2	100±0	63±3±4
4		0	60±1	31±4		32±8	
5		4±10	74±5	24±12		63±2	52±4±0
6		67±2	45±9			9±6	
7		17±9	24±15			5±2	
8		68±3	8±5*			65±4*	
9		54±10	16±4			29±1	
10		6±4	22±5			30±8	
11		56±2	46±2			69±1	38±1

\*Potentiometric method.

Trypsin activity was measured by the electrometric method using a multi-channel unit. N-benzoyl-L-arginine methyl ester hydrochloride in an end concentration of  $1 \cdot 10^{-3}$  M was used as substrate, and brand B bovine trypsin, with specific activity of 18 units/mg served as the enzyme. The reaction was run at medium pH of 7.8 produced by means of tris-HCl buffer in an end concentration of  $5 \cdot 10^{-3}$  M. In order to determine ACE and BuCE activity, we used mainly the spectrophotometric method of Ellman [10], in which acetylthiocholine bromide in a concentration of  $5 \cdot 10^{-4}$  M served as the substrate and purified human ACE preparations with specific activity of 1.2 units/mg or BuCE with specific activity of 5.9 units/mg served as the enzyme. In some cases, we used potentiometric titration at a constant pH [13]. The end concentration of aminoesters used in the incubation mixture constituted  $1 \cdot 10^{-3}$ - $1 \cdot 10^{-5}$  M. With all of the tested compounds, the inhibitory effect on enzymes (if it was demonstrable) was reversible and did not increase in the course of incubation. The only exception was compound 1, in the course of incubation of which with BuCE we observed some increase in inhibitory effect with time. For this reason, compound 1 may be considered as a quasi-substrate, which binds with the active center and is harder to hydrolyze and deacylate by the enzyme.

Aminoesters of the cyclohexene series (1, 2, 4, 6) were obtained according to [11]; 3-[1-(1-cyclohexenyl) ethoxy]propylamine (compound 7) was obtained according to [12].

Trimethyl-1-(1-cyclohexenyl)ethoxycarbonyl methylammonium iodide (3): A mixture of 1-(1-cyclohexenyl) ethyl-N-dimethylaminoacetate and methyl iodide (1:5) in ethyl alcohol was boiled in a round-bottom flask with reflux condenser for 4 h. The sediment was recrystallized from alcohol; melting point 221°C. The following were demonstrated: 31.94% C, 6.79% H, 35.80% I, 3.88% N.  $C_{13}H_{25}NO_2I$ . The calculated figures were: C 44.19%, 6.84% H, 35.94% I and 3.96% N.

Methyl-1-(1-cyclohexenyl) ethoxycarbonyl methylpiperidinium iodide (5) was obtained just like the preceding compound, melting point 118-120°C. The following were demonstrated: 48.94% C, 7.18% H, 32.20% I and 3.61% N.  $C_{16}H_{28}NO_2I$ . The calculated figures were: 48.86% C, 7.17% H, 32.29% I and 3.56% N.

(2-Cyclohexenyl) oxycarbonyl methylbenzylmorpholinium chloride (8) and (2-cyclohexenyl) oxycarbonyl methyl(2-cyclohexenyl) morpholinium chloride (9) were obtained by warming equimolar amounts of 2-cyclohexenyl morpholinoacetate with benzyl chloride (compound 8) and 3-chlorocyclohexene-1 (compound 9). Melting point of compound 8 is 238-239°C (from alcohol). For compound 9 the melting point is 218-219°C (from a mixture of ethyl acetate and alcohol).

(2-Cyclohexenyl) oxycarbonyl propylamine (10): To suspension of lithium aluminohydrode in absolute ether, we slowly added (2-cyclohexenyl) oxycarbonyl propionitrile in ether under refrigeration. After addition of all of the nitrile, the reaction mixture was stirred for 5 h at room temperature. We then carefully separated nonreacting LiAlH<sub>4</sub> with 40% aqueous KOH. The ether extracts were dried over MgSO<sub>4</sub>. The end product was isolated by vacuum distillation; boiling point is 84°C/4 mm,  $n_D^{20}$  1.4840.

3-(2-Cyclohexenyl) oxypropyl trioxyethylammonium chloride (11) was obtained by heating in a vial of a reaction mixture containing 0.043 mole 3-(2-cyclohexenyl) oxypropionitrile and 0.1 mole ethylene chlorohydrin for 15 h; melting point 243-245°C; C<sub>16</sub>H<sub>36</sub>NO<sub>4</sub>Cl.

The studies described here are important to the search and purposeful synthesis of compounds that have selective effects on specific enzymes, which catalyze the most important physiological processes.

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[161-10,657]

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CSO: 1841

UDC 557.11

VENOM OF ANCISTRODON BLOMHOFFII USSURIENSIS: I. GENERAL CHARACTERIZATION OF WHOLE VENOM

Tashkent KHIMIYA PRIRODNYKH SOYEDINENIY in Russian No 1, 1980 pp 133-134 manuscript received 3 Oct 79

SADYKOV, E. S., IKRAMOVA, S. I., BARABANSHCHIKOVA, N. A. and YUKEL'SON, L. Ya., Uzbek SSR Academy of Sciences Institute of Biochemistry, Tashkent

[Abstract] The results are given of a study providing a preliminary characterization of the composition, properties and effect of the whole venom of *Ancistrodon blomhoffii ussuriensis* of the Crotalidae family. The ultimate purpose of the study was to produce from the venom pure biologically active substances. A determination by the Lowry method and an analysis of absorption spectra of aqueous solutions of the venom demonstrated that as much as 80 percent of the entire mass of the venom is made up of substances of a protein nature. Among the venom's proteins predominate components with a molecular weight greater than 20,000. Electrophoresis demonstrated that acidic or neutral proteins predominate in the composition. In the subcutaneous administration of the venom to mice was caused the formation of a hemorrhagic zone whose area was proportional to the concentration of the venom. In small doses up to 100  $\mu$ g the venom stimulated the formation of a fibrinous clot in the citrated plasma, and in higher concentrations it had an anticoagulating effect and caused fibrinolysis. The venom does not possess direct hemolytic activity but lyses washed erythrocytes in the presence of exogenous phospholipids. The latter fact can testify to the presence of phospholipase  $A_2$  in it. The definite presence of phospholipase  $A_2$  was proven by direct measurements by means of the titrometric method and by the inhibition of the coagulation of an egg yolk. In addition, in the venom were found proteases hydrolyzing casein and hemoglobin and various phosphatases, such as nonspecific phosphoric and phospho diesterases, 5-nucleotidase, nucleases and ATP-pyrophosphatase. Fractionation of the venom is planned in a future study. References 7: 4 Russian, 3 Western.

[145-8831]

UDC 577.11

COMPARATIVE INVESTIGATION OF THE HEMOLYTIC EFFECT OF TWO CYTOTOXINS FROM THE VENOM OF THE CENTRAL ASIAN COBRA NAJA OXIANA EICHWALD

Tashkent KHIMIYA PRIRODNYKH SOYEDINENIY in Russian No 1, 1980 pp 82-84 manuscript received 3 Oct 79

KKHOLE, V., SADYKOV, E. S., YUKEL'SON, L. Ya. and TASHMUKHAMEDOV, B. A., Uzbek SSR Academy of Sciences Institute of Biochemistry, Tashkent

[Abstract] Cytotoxins, found in the venom of Elapidae, cause cell lysis, which is intensified with the addition of  $A_2$  phospholipases. The results are given of a comparative investigation of the hemolytic effect of two cytotoxins,  $V_{c1}$  and  $V_{c5}$ , separated in pure form from the venom of the Central Asian cobra Naja oxiana Eichwald. Cytotoxin  $V_{c1}$  is distinguished by a lower content of lysine and a higher content of dicarboxylic amino acid residues, which is responsible for the lower basicity of its molecules, which has been established by electrophoresis. The present study was carried out in order to assess the significance of a positive charge of the molecules of both cytotoxins in the mechanism of their influence on membranes. It is demonstrated that with overall considerable similarity in the kinetics of their direct hemolytic effect, cytotoxin  $V_{c5}$  is characterized by greater hemolytic effectiveness than  $V_{c1}$ . The calculated values of  $HD_{50}$  of cytotoxins  $V_{c1}$  and  $V_{c5}$  equaled 2000 and 1400  $\mu g$ , respectively. The lytic effect of both cytotoxins evidenced a similar dependence on the pH of the medium. With an increase in the absolute pH of the medium, when ionization of negatively charged groups of the membrane increases the interaction of cytotoxins with the membrane is intensified and the more basic cytotoxin  $V_{c5}$  lyses erythrocytes more actively than does cytotoxin  $V_{c1}$ . A gradual increase in the direct hemolytic effect is evidenced with a rise in temperature to 30°C, and with a further rise in the incubation temperature the hemolytic effect of the cytotoxins is intensified drastically. The higher hemolytic effectiveness of cytotoxin  $V_{c5}$  correlates with the greater basicity of its molecules. This correlation makes it possible to confirm the significance of a positive charge of cytotoxin molecules in the mechanism of their lytic effect. This conclusion confirms earlier hypotheses based on an analysis of the structure of cytotoxins and their interaction with artificial phospholipid membranes.

Figures 2; references 10: 5 Russian, 5 Western.

[145-8831]

UDC 591.145:598.126

INVESTIGATION OF THE VENOM OF THE STEPPE ADDER VIPERA URSPINI RENARDI: II. CHEMICAL AND BIOLOGICAL CHARACTERISTICS OF FRACTIONS

Tashkent KHIMIYA PRIRODNYKH SOYEDINENIY in Russian No 1, 1980 pp 73-76 manuscript received 21 Sep 79

LYUBIMTSEVA, G. Ye. and YUKEL'SON, L. Ya., Uzbek SSR Academy of Sciences Institute of Biochemistry, Tashkent

[Abstract] The results are given of the fractionation of the venom of the steppe adder Vipera ursini renardi Ch. by employing "Sefadex G-75," and of a study of the

chemical properties and biological effect of the fractions obtained, as compared with whole venom. Five fractions were produced by passing the whole venom through the gel. Fractions I, II, III, IV and V had a yield, respectively, of 29.2, 23.2, 22.1, 5.1 and 18.1 percent and a protein content of 92.0, 93.0, 98.0, 37.0 and 37.0 percent. Proteolytic activity was discovered primarily in fractions I and II. Phospholipase A<sub>2</sub> is separated from the proteolytic enzymes and is included in the composition of fraction III. These three fractions make up the main mass of the whole venom. A comparative table is given of the results of investigations of the enzymatic activity and biological effect of the whole venom of the steppe adder and of fractions produced from it. Data are presented on toxicity, protease, fibrinolytic activity, phospholipase, indirect hemolysis, coagulating activity and hemorrhagic and lethal effect, and the third fraction was identified as phospholipase A<sub>2</sub>. Toxicity was determined by intraperitoneal administration of the material to white mice weighing 18 to 20 g. The value of LD<sub>50</sub> and its upper and lower confidence limits were calculated according to Litchfield and Wilkinson. The hemorrhagic effect was estimated according to the Kondo method (1960) 30 min after introduction of the material in a dosage of 2.5 mg/kg. Proteolytic activity was determined by the Kunitz method (1947) employing a casein substrate, phospholipase activity was determined according to the time for the inhibition of coagulation of an egg yolk, and coagulating activity in relation to the coagulation of human citrated plasma. For fractionation was used a column of "Sefadex G-75" measuring 25 X 850 mm and elution was carried out with a 0.05 M solution of ammonium bicarbonate at a rate of 30 mm/h. Proteolytic and phospholipase activity was determined in specimens. Protein "peaks" were collected, concentrated, desalinated by passing through "Sefadex G-25" and desiccated by lyophilization. Disk electrophoresis was carried out in a 7.5 percent solution of polyacrylamide gel in buffer systems with a pH of 4.3 and 8.3. On the basis of the experiments the hypothesis is offered that the coagulating and hemorrhagic activity of the venom of the steppe adder are related primarily to the activity of proteolytic enzymes. Figures 1; references 16: 7 Russian, 9 Western. [145-8831]

UDC 615.281.547.73

#### BIOLOGICALLY ACTIVE DERIVATIVES OF THIOPHENE. 2. SYNTHESIS AND BIOLOGICAL ACTIVITY OF SOME THIOPHENE UNSATURATED KETONES AND THEIR DERIVATIVES

Moscow KHIMIKO-FARMATSEVTICHESKIY ZHURNAL in Russian No 4, 1980 submitted 13 Jul 79 signed to press 27 Mar 80 pp 36-39

KULIKOVA, D. A., CHURKIN, Yu. D., and PANIFILOVA, L. V., Scientific Research Institute of Biological Tests of Chemical Compounds, Moscow Oblast

[Abstract] Some new alpha, beta--unsaturated ketones of the thiophene series and some of their derivatives (semicarbazones, thiosemicarbazones, oximes phenylhydrazones, 2,4-dinitrophenylhydrazones, were synthesized and their antimicrobial effect was studied. It was found that all of these compounds produced a pronounced antimicrobial effect and many of them produced a pronounced antifungal effect. The structures of the synthesized compounds proved to be decisive in their effect. The position of the NO<sub>2</sub> group in the thiophene ring was decisive for antibacterial

activity while not the position but the presence of the  $\text{NO}_2$ -group with a short side chain was decisive for antifungal activity. References 7:  
[183-2791]

UDC 615.31:547.873

#### SYNTHESIS AND SOME PHARMACOLOGICAL PROPERTIES OF ISOFERVENULIN DERIVATIVE

Moscow KHMIMKO-FARMATSEVTICHESKIY ZHURNAL in Russian No 4, 1980 submitted 23 Jul 79  
signed to press 27 Mar 80 pp 39-44

AZEV, Yu. A., POSMOVSKIY, I. YA., PIDYEMSKIY, E. L. and GOLENEVA, A. F., Ural'sk Polytechnical Institute imeni S. M. Kirov, Sverdlovsk

[Abstract] Corresponding 3-alkylsulfonyl derivatives were produced by oxidation of 3-alkylthiopyrimido [4.5-e] [1.2.4] triazine-6,8-diones and analogs of the antibiotic fervenulin was produced by substitution of the 3-alkylsulfonyl group in the pyrimido [4.5-e] [1.2.4] triazone-6,8-diones by different nucleophils. A pharmacological study of the anti-inflammation, analgesic and anti-microbial properties of these derivatives following intraperitoneal injection into Wistar rats and white mice indicated that their low toxicity and the rather high phlogolytic activity of some of them justifies the further study of them as effective anti-inflammation agents. References: 7. Figure 1.

[183-2791]

UDC 615.31:547.78].015.11

#### BIOLOGICAL ACTIVITY OF AZOLIDONES AS A FUNCTION OF STRUCTURE

Moscow KHMIMKO-FARMATSEVTICHESKIY ZHURNAL in Russian No 4, 1980 submitted 1 Apr 79 signed to press 27 Mar 80 pp 44-49

LEBEDEV, R. S. and LEBEDEVA, I. R., Bryansk Pedagogical Institute imeni I. G. Petrovskiy

[Abstract] A study of the regularities of change of biological activity of azolidones during heterosubstitution showed a quantitative connection of the biological activity with parameters which characterize the structure of the azolidones and factors. The center of activity was found in the substructure SONH. Small oscillation of azolidone molecules were found to provide basic differences of biological activity and reaction capacity during heterosubstitution. The high correlation of spectral properties, non-spectral properties and biological activity of the heterocycles regardless of features of the environment indicated the possibility of production effective biological activity preparations based on structural variations of azolidones molecules. References 16, Figure 1.  
[183-2791]

## POLYMERS AND POLYMERIZATION

UDC 547.458.81:66.085.3

### FEATURES OF GRAFT POLYMERIZATION DURING THE THAWING OF CELLULOSE-STYRENE SYSTEM GAMMA-IRRADIATED AT -196°C

Riga KHIMIYA DREVESINY in Russian No 2, 1980 pp 113-114 manuscript received  
29 Nov 79

PLOTNIKOV, O. V., LESIN'SH, A. Ya. and RAYABEYE, E. L., Institute of Wood Chemistry,  
Latvian SSR Academy of Sciences

[Abstract] The pattern of graft polymerization during the thawing of a cellulose-water-styrene system subjected to gamma-irradiation at liquid-nitrogen temperature was investigated by EPR spectra and scanning calorimetry. The EPR spectra of the gamma-irradiated specimens were found to consist of five lines characteristic of cellulose macroradicals. Thawing of the specimens resulted in the destruction of the macroradicals located in the parts of cellulose accessible to water molecules. The macroradicals located in crystalline regions survive the temperature range within which the peak heat release associated with polymerization takes place. This is in agreement with the earlier finding that graft polymerization is intensified when the more crystalline specimens of cellulose are used. According to EPR and calorimetric data, following the melting of styrene (m.p. -33°C), a liquid monomer and macroradicals of cellulose are present in the system, but the process of polymerization commences at ~10°C when, apparently, sufficient mobility of cellulose macroradicals is reached. The activation energy of polymerization (~80 kjoules/mole) exceeds by a factor of more than 2 of the activation energy of chain growth for most vinyl polymers. This indicates that the process is limited by diffusion. Figure 1; references 5: 3 Russian, 2 Western.  
[160-1386]

UDC 661.7:547.292'361.3

### INDUSTRIAL SYNTHESIS OF ALLYLACETATE FROM PROPYLENE AND ACETIC ACID

Moscow KHMICHESKAYA PROMYSHLENNOST' in Russian No 5, 1980 signed to press  
29 Apr 80 pp 275-276

KHCHEYAN, Kh. Ye., FEDOROVA, N. M., DARMAN'YAN, P. M., SAMTER, L. N., MAK, N. Ye.,  
ZAVOROTOV, V. I., PINKHASIK, E. V., KREKHOVA, Z. G. and SHURUPOVA, L. N.

[Abstract] A process of production of allylacetate from propylene and acetic acid, the separation of the specific product from the condensate and regeneration of the

catalyst which has been worked out completely on an experimental device and is being mastered industrially was described and discussed. A continuous method for conducting the industrial process with recovery of the unreacted gases and regeneration of the catalyst was described. The method is distinguished by its simplicity, high selectivity, availability of feed stock, almost complete absence of waste waters and high economic effectiveness. References 6; Figure 1.  
[214-2791]

UDC 661.715.352.4; 661.725.4; 661.727.6; 661.7:  
547.422.24; 661.7:547.361.3; 661.7:547.384.4

#### SOME COMPOUNDS SYNTHESIZED ON THE BASIS OF BUTENE-2 OXIDE

Moscow KHIMICHESKAYA PROMYSHLENNOST' in Russian No 3, 1980 pp 142-144

STEPANOVA, I. P., OBUKHOV, V. M., BONDARENKO, A. V. and FARBEROV, M. I.

[Abstract] The commercial production from butene-2 oxide of secondary butyl alcohol, methylethyl ketone, butadiene-2,3, methylvinyl carbinol, methylvinyl ketone and butadiene-1,3 is examined. The products are important reagents in the chemical industry for the production of solvents, plasticizers, surfactants and polymerizers. Reaction conditions are explained and a schematic diagram of a commercial butadiene-1,3 plant is given. The examined processes are high-yield reactions that proceed in commercial reactors under routine industrial conditions. Figures 3; tables 2; references: 9 Russian.  
[158-7872]

UDC 677.494.743.21-96

#### PRODUCTION AND PROPERTIES OF FIBERS BASED ON POLYVINYLIDENE FLUORIDE-TETRAFLUORETHYLENE COPOLYMER

Moscow KHIMICHESKIYE VOLOKNA in Russian No 2, 1980 pp 44-45 manuscript received  
17 Apr 79

VOL'F, L. A., BEZPROZVANNYKH, A. V., LOGVINOV, A. I., VESAYTE, B. S.,  
MADORSKAYA, L. Ya., LOGINOVA, N. N. and PANSHIN, Yu. A.

[Abstract] Results are presented of an investigation of a process whereby fibers are produced from a copolymer of polyvinylidene fluoride and tetrafluorethylene, containing up to 5% tetrafluorethylene, which is soluble in aliphatic ketones and substituted organic acid amides. Acetone is used for making spinning solutions. Fibers are spun wet from 23% acetone solutions in water-acetone precipitators. The physicomechanical properties of the fibers are as good as when spun in water-acetone baths and have high resistance to corrosive chemicals and heat. The new fiber is stronger than tetrafluorethylene-base fiber and is 30-50 times better than all known fluorine-base fibers in terms of bending strength. Figures 2; tables 2; references: 9 Russian.  
[155-7872]

UDC 677.462.044.17

MTILON FIBER PRODUCTION WITH REVERSIBLE REDOX SYSTEM

Moscow KHMICHESKIYE VOLOKNA in Russian No 2, 1980 pp 36-37 manuscript received 16 Jan 79

VOINOVA, G. Yu., BREUSOVA, I. P., MORIN, B. P. and STANCHENKO, G. I.

[Abstract] The  $\text{Fe}^{3+}$ - $\text{H}_2\text{O}_2$ -hydrazine reversible redox system is proposed as a means of satisfying the growing public demand for mtilon fiber. The proposed system would produce grafted cellulose copolymers at a higher rate and at a lower temperature than previously suggested methods and the product would have the necessary concentration of graft polymer. The production process would proceed in a semicontinuous, instead of batch reactor, in which the technological solutions used for treating the fiber with iron ions would be recycled. Tables 2; references 5: 4 Russian, 1 Western.

[155-7872]

UDC 541.64:547.538.141

ACETYL HALIDE DERIVATIVES OF COPOLYMERS OF STYRENE AND DIVINYLBENZENE AS ENZYME IMMOBILIZATION CARRIERS

Moscow VYSOKOMOLEKULYARNYYE SOYEDINENIYE in Russian No 4, 1980 pp 789-793 manuscript received 31 Oct 78

MAKAROVA, S.B., GRYAZNOV, G. V., LITVAK, Zh. M., PROSVETOVA, N. K., MENDEL'SON, Ye. A., SHABANOVA, N. V. and KIM, G. I.

[Abstract] Acetyl halide derivatives of copolymers of styrene and divinylbenzene were synthesized and analyzed as enzyme immobilization carriers for the purpose of obtaining synthetic polymers with high mechanical strength and with a polymer framework with controllable permeability. An analysis of the IR-spectra of the analyzed acetyl bromide derivatives of copolymers with different macromolecular structures and of the influence of the macromolecular copolymer structure on the acetyl bromination process shows that the Friedel-Crafts reaction proceeds rather readily in polymer lattices, regardless of the degree of cross linking. The substitution of bromine by iodine by reacting acetyl bromide derivative copolymers with potassium iodide in ethyl alcohol produces acetyl iodide derivative copolymers. The reaction proceeds to completion and the iodine concentration in the copolymers is 30-60%, depending on the bromine concentration in the original compounds. Acetyl iodide derivative copolymers of styrene and divinylbenzene with a high iodine concentration in the macroporous structure are found to be best suited for immobilizing the enzymes glucoamylase and pepsine. Figures 1; tables 3; references 10: 7 Russian, 3 Western.

[157-7872]

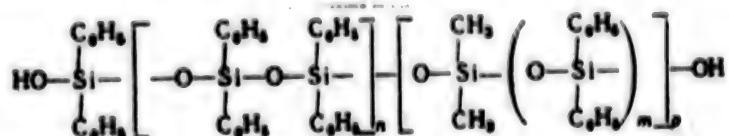
UDC 678.84:66.095.3

SECONDARY REACTIONS DURING THE SYNTHESIS OF  $\alpha$ ,  $\omega$ -DIHYDROXYPOLYDIMETHYLPHENYL-SILOXANE

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 13-14

MAL'NOVA, G. N., IVANOVA, N. A., SKOROCHODOV, I. I., BABUSHKINA, S. A., ZHINKIN, D. Ya., and BACHKAREVA, S. V.

[Abstract] The synthesis of the title polymer (compound I) via the polycondensation of hexamethylcyclotrisilazane (HMCTS) with diphenylsilanediol (DPSD) may be complicated by several side reactions, such as 1) polycondensation of only the DPSD; 2) reaction of compound (I) with HMCTS, leading to the formation of a polymer having the following structure



3) reaction of the terminal -OH group on the growing polymer of (I) which limits its reactivity and results in a lower average molecular weight for the product. A batch of polymer (I) was prepared using the standard method and separated into 12 fractions using an acetone-water system. With increasing fraction number, the % concentration, the viscosity, and relative viscosity in toluene, and the average molecular weight decreased. Side reactions may be suppressed by maintaining low concentrations of ammonia and hydrosilicate in the reacting medium. Tables 1; references: 2 Russian.

[193-12027]

UDC 678.746.22:620.178.7:541.12.03

RHEOLOGICAL AND SORPTION PROPERTIES OF IMPACT-RESISTANT POLYSTYRENE

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 15-16 manuscript received 2 Jun 78

ASHUROV, N. R., BOLOTNIKOVA, L. S., KALYUZHNAIA, L. M., LISHANSKIY, I. S., NESTEROV, V. V., PANOV, Yu. N., and FREN'KEL, S. Ya.

[Abstract] Impact-resistant polystyrene (IRP) can be considered a two-phase colloid of rubber-like material dispersed in a glass matrix. Five samples were evaluated for parameters such as % concentration of rubber-like material, mass average molecular weight of the matrix, diameter of the dispersed-phase particles, etc. For all the samples, the rate of shear at a given viscosity was higher than that for the matrix; this effect becomes more pronounced with increasing concentration of rubber-like material in the IRP. This and other observed effects may be explained by the formation in the IRP of an additional network whose junctions are formed by particles of the dispersed phase. Under shear tension, this network is easily broken down at which point the rheological properties of the system are no different from

those of the matrix. The dynamic modulus of elasticity increases with increasing concentration of rubber-like material. The presence of the matrix also influences the adsorption capabilities, the latter decreasing with increasing concentrations of the rubber-like material. This and other observations can apparently be explained by the presence of residual rubber-like material. Figures 2; tables 1; references 12: 9 Western, 3 Russian.

[193-12027]

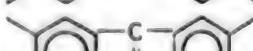
UDC 678.675.4 01:539.3

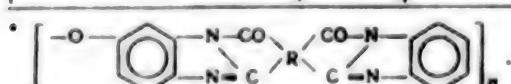
### PROPERTIES OF MATERIALS BASED ON POLYBENZIMIDAZOPYRROLONE

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 16-18 manuscript received 26 Jun 78

KORSHAK, L. L., LEKAYE, I. A., VINOGRADOVA, O. V., CHATOVA, L. L., LEKAYE, T. V., and RUSANOV, A. L.

**[Abstract]** Polymers based on polyheteroarylene compounds are characterized by high radiation and ablation resistance and are prepared by a two-stage synthesis: preparation of the soluble polyamidoamino acids (PAAA's), and polycondensation by thermal intramolecular polycyclodehydration of the PAAA's. Three types of polymers were prepared by the reaction in dimethylformamide of 3,3',4,4'-tetraminodiphenyl-oxide with the dianhydrides of diphenyloxide-, benzophenone-, and diphenylsulfon-tetracarbonic acids and pyromellitic acid. An evaluation was made of the optimum regimes for extruding these polymers and of the properties of the extruded material. The table below summarizes the latter (where A = impact velocity; B = breaking point during bending; C = Brinell hardness; and D = density)

	Ударная вязкость, кДж/м <sup>2</sup>	Разрушающее из- жение при изгибе, МПа	Твердость по Бриннелю, МПа	Плотность, г/см <sup>3</sup>
(A)	(B)	(C)	(D)	
	37.0	>150	650	1.33
	4.0	>150	610	1.35
	3.0	23	550	1.29
	1.5	40	810	1.28



Figures 3; references 9: 5 Western, 4 Russian  
[193-12027]

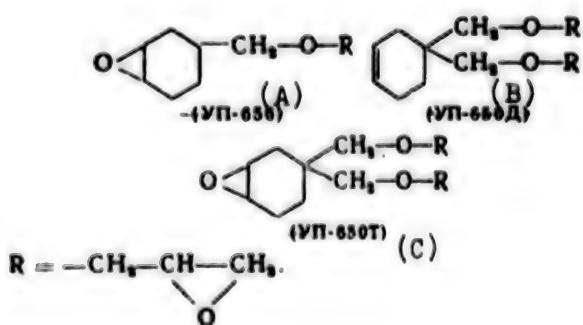
UDC 678.686

## POLYMER MATERIALS BASED ON ALIPHATIC-CYCLOALIPHATIC EPOXIDE COMPOUNDS

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 18-19 manuscript received  
26 Sep 78

PET'KO, I. P., BEYDA, V. I., BATOG, A. Ye., STEPKO, O. P., and PANDAZI, I. F.

[Abstract] The title study was carried out using the following aliphatic-cycloaliphatic epoxide compounds (ACAEC):



Measurements and evaluations of their physical properties indicate that the latter are determined to a significant degree by curing agent. The use of the p-isomer of 4,4'-diaminodiphenylsulfone (DADPS), 4,4'-diaminodiphenylmethane, or p-aminobenzylalanine permitted the condensation of polymers having better properties than those obtained from the o-isomer of 3,3'-DADPS. Aromatic amines are probably the best curing agents for ACAEC's containing more than two glycidic groups. Polymers based on monomers containing three epoxide groups of type C (in the table) have more valuable properties than those based on compounds having only two, such as A and B. In addition, the physical and mechanical properties of polymers containing only two epoxide groups, such as A and B are inferior. Tables 3; references: 2 Russian.

[103-12027]

UDC 678.746.45 01:678.046

## PROPERTIES OF COMPOSITE MATERIALS BASED ON POLYPHENYLENOXIDES PLUS TITANIUM DIOXIDE

Moscow PLASTICHESKIE MASSY in Russian No 5, 1980 pp 23-24

PANOVA, L. M., KOZLOVZ, Z. F., GLADKAYA, T. G., and YUDKIN, B. I.

[Abstract] Polymers doped with inorganic dielectric materials have found applications in many fields such as radio and electronics technology and instrument design. The dielectric properties of the title system were evaluated for such use.

Six samples of the composite containing from 1.3 to 38% TiO<sub>2</sub> were synthesized. The dielectric permeability ranged from 2.6 at 10<sup>7</sup>hz for 1.3% TiO<sub>2</sub> to 11.0 at 10<sup>3</sup>hz for 38% TiO<sub>2</sub>. The permeability increased slightly with decreasing frequency and strongly with increasing TiO<sub>2</sub>. The dielectric permeability changed in a rather erratic fashion when the samples were exposed to a 98% humidity environment. Generally, the most significant changes were observed for samples containing 38% TiO<sub>2</sub> at all frequencies; the least change was observed at 10<sup>7</sup>hz for the low range TiO<sub>2</sub> concentrations. With increasing exposure time, the dielectric permeability of samples containing high TiO<sub>2</sub> increased while that for samples having low %TiO<sub>2</sub> either remained the same or decreased. This material appears to be suited for the suggested applications. Tables 3; Figures 2.

[193-12027]

UDC 547.568.5:678.643'42'5

#### MODIFICATION OF THE PHENOLALCOHOLS OF WATER-SOLUBLE EPOXIDE RESINS

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 31-32 manuscript received 25 Jun 78

LASTAUSKENE, G. B., GRIBAUSKENE, E. L., and DAMBRAUSKAS, L. P.

[Abstract] Due to the presence of a large number of reactive epoxide groups (20-26%), the water-soluble aliphatic epoxide resins may react with hydroxyl, carboxyl, and other functional groups. A sample was synthesized by mixing the phenolalcohols with acetone solutions of the water-soluble epoxide resins to which some maleic anhydride had been added. The components mixed easily and the mixture did not lose its reactivity when mixed with water. The physical and chemical properties of the cured resin samples were superior to and not linearly related to those of the starting materials. The breaking point of the new material decreased by only 10% during a seven-day exposure in a humid environment, whereas the starting materials showed a decrease of over 50%. An infrared scan showed no adsorption bands which could be assigned either to the maleic acid or to the starting materials. Hence a chemical reaction had occurred among all the reactants. Figures 3; references: 5 Russian.

[193-12027]

UDC 678.128.6:678.01:53

#### INFLUENCE OF MULTIPLE PROCESSING ON THE PROPERTIES OF POLYMERS

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 40-41 manuscript received 5 Jul 78

MOSKATOV, K. A.

[Abstract] Plastics materials are increasingly being recycled, reprocessed and then combined with virgin stock in various proportions. Normal reprocessing,

however, causes a deterioration in the properties of the plastics. A polyamide showed the following change in desirable characteristics after having passed through two additional processing cycles: decrease in molecular weight from 13,350 to 10,260; a decrease in the specific viscosity from greater than 67 to greater than 26; and a decrease in the breaking point from 56 to 36. Heat processing, however, can increase the strength indicators by approximately 20%; thus polyamides can be reprocessed up to seven times without significant deterioration of desirable properties. Manufactured polystyrene articles were subjected to 20 cycles of reprocessing, resulting in an increase in the viscosity of the melt from less than 10 to 30 (at 473°K) and then to about 44 (at 513°K). The molecular weight decreased from 280,000 to 140,000. Five reprocessings is the maximum for polystyrene to retain its desirable properties. For low pressure polyethylene, a sample containing recycled material retains the properties of virgin material if the mixture contains less than 16% doubly processed granules, less than 3.2% triply processed granules, or less than 0.64% quadruply processed granules. The factor most sensitive to change during reprocessing is the molecular weight. The minimum acceptable molecular weight for polyamides is 10,000; for polystyrol, 200,000; and for low pressure polyethylene, 350,000. Figures 2; references 8: 6 Western, 2 Russian. [193-12027]

UDC 678.742.2 01

#### POLYETHYLENE GRADE TYPES ADOPTED BY CEMA MEMBER COUNTRIES AND THE PROSPECTS FOR THEIR DEVELOPMENT

Moscow PLASTICHESKIYE MASSY in Russian No 5, 1980 pp 53-55

ANDREYEVA, I. N., GRIGOR'YEV, V. A., KARASEV, A. N., and POLYAKOV, A. V.

[Abstract] A catalogue has been compiled of the grade and characteristic descriptions of low pressure polyethylene, which is marketed under the following names: bulen in the Republic of Bulgaria, polyethylene shkopau in East Germany, polyethylene ND (PEND) in the USSR and liten in Czechoslovakia. PEND is prepared by the polymerization of ethylene over complex organometallic catalysts at temperatures lower than 433°K and pressures higher than 6 MPa. The physical properties of the sample are a function of the catalyst, the phase composition, and the medium used during polymerization (in suspension, solution, or gas phase). The grade designations, properties, and in some cases remarks on the synthesis are given in separate tables for each of the five "families" of polyethylenes. Tables 5. [193-12027]

UDC 678.01:539.539.612

THE EFFECT OF THE CHEMICAL STRUCTURE OF POLYMERS ON THEIR ADHESIVE STRENGTH

Moscow KOLLOIDNYY ZHURNAL in Russian Vol 42, No 3, May-Jun 80 pp 458-463 manuscript received 20 Apr 79

GUL', V. Ye., RYBALOVA, G. V., ZAVIN, B. G., et al., Moscow Technological Institute of Meat and Dairy Industry, Institute of Metal Organic Compounds, USSR Academy of Sciences, Moscow

[Abstract] The aim of this study was to analyze factors responsible for minimal adhesion between wrapping materials and protein products. The study included the principal functional groups responsible for adhesive bonds: the polar groups -OH, -COOH, -NH<sub>2</sub>, -SH, SS-, CO-NH, etc. Collagen and myosin were selected as models representing protein products. Specific adhesive energies of polyorganosiloxane coatings and proteins were analyzed. As the content of diphenylsiloxane links in the polyorganosiloxanes increased, the antiadhesive properties of the wrapping material decreased. This was accompanied by a decrease in the difference between the specific adhesion energy of the proteins and of the polyorganosiloxane coatings. Figures 4; references 12: 6 Russian, 6 Western.  
[212-7813]

UDC 678.643'42'5

CHARACTERISTICS OF PRODUCTION AND PROPERTIES OF METALLOPOLYMERS BASED ON INTER-POLYMER SYSTEMS

Kiev KHIMICHESKAYA TEKHNOLOGIYA in Russian No 3, 1980 submitted 10 Aug 78 signed to press 7 May 80 pp 26-29

IL'YINA, Z. T.

[Abstract] A study of the principles of formation and of the properties of metallopolymers based on polymer mixtures was described and some features which distinguish these systems from metallocopolymers based on individual polymers were discussed. It was found that a basic peculiarity of metallocopolymers based on inter-polymer systems is that not only the presence of compatibility between the polymer components is important but also the mechanism of compatibility and especially the intensity of interaction between the macromolecules. A second peculiarity is the abrupt growth and the extreme dependence of the thermomechanical and strength properties of metallocopolymers on the content in them of metal. Some electron microscopic findings concerning the structure of such metallocopolymer systems were discussed briefly. References 15: Figures 5.  
[211-2791]

UDC 678.742.2.046

THE EFFECT OF FILLERS ON PROPERTIES OF POLYETHYLENE

Kiev KHMICHESKAYA TEKHNOLOGIYA in Russian No 3, 1980 submitted 10 Jan 79 signed to press 7 May 80 pp 23-25

IL'YENKO, R. Ye. and GORDIYENKO, V. P., UkrSSR Academy of Sciences Institute of Physical Chemistry

[Abstract] A study of the properties of polyethylene containing different kinds of fillers in a wide range of concentrations (up to 17.8% by volume) with the use of high density polyethylene mark 21006-075 and low density polyethylene mark 15802-020 and 150303-003 and the use of aerosols mark A-175, titanium dioxide mark P-02 and chopped fiberglass NSO-6 (5-6 mm) as fillers introduced into the polymer melt at 160-180°C, indicated that low concentrations of dispersed fillers lead to a pronounced increase of the Vitk softening point and Brinell hardness and some increase of relative elongation and the breaking point of both high density polyethylene and low density polyethylene. This is probably due to an increase of the structural ordering of the crystallizing polymer and reduction of secondary supermolecular formations. A knowledge of the nature of change of properties of compositions based on polyethylene with different kinds of fillers makes possible production of composite material with a fixed complex of properties. References 5; Figures 5.  
[211-2791]

UDC 541.49:547.87+678.643.425

CUPROAMMONIACAL (AMINE) SALTS OF CYANURIC ACID AND DIALLYLISOCYANURIC ACID AS FIRE-INHIBITING ADDITIVES

Yerevan ARMYANSKIY KHMICHESKIY ZHURNAL in Russian No 4, 1980 submitted 4 Apr 79 signed to press 30 Apr 80 pp 290-294

YESAYAN, G. T., KAZARYAN, S. M. and ORDYAN, M. B., Armenian SSR Academy of Sciences Institute of Organic Chemistry, Yerevan State Medical Institute

[Abstract] Corresponding complex compounds produced by interaction of copper sulfate with aqueous solutions of ammonia, mono- and diethanolamine salts of diallylisocyanurate were obtained and their effect and the effect of previously synthesized analogous complexes of cyanuric acid on combustibility of ED-20 epoxy resins hardened by 4,4'-diaminodiphenylmethane were studied. It was found that such modification of ED-20 epoxy resins increased their fire resistance as characterized by the oxygen index in comparison to the inflammability of unmodified resins. Diallylisocyanurate complexes are most miscible with the resin but provide a lower oxygen index and require a higher level of metal than do cyanuric acid complexes. References 9; Figure 1.  
[213-2791]

UDC 66.067:[661.7:547.466.3-318]

HYDROGENATION OF AQUEOUS SOLUTIONS OF CAPROLACTAM IN A FIXED CATALYST BED

Moscow KHIMICHESKAYA PROMYSHLENNOST' in Russian No 6, 1980 pp 341-342

IOSELIANI, E. G., YURSHA, I. A. and LEVCHENKOV, A. N.

[Abstract] The purification of aqueous solutions of caprolactam by hydrogenation of unsaturated microimpurities accompanying the monomer is usually performed by using an aqueous suspension of Raney nickel catalyst (particle size < 100  $\mu$ ) under a hydrogen pressure of 5-6 kg/cm<sup>2</sup> at 90°C. Since this complicates transportation, proportioning, and subsequent filtration, the possibility of using the Raney nickel catalyst in a fixed-bed form was experimentally investigated with the aid of a specially designed experimental reactor, with the fixed bed consisting of a bottom layer of gravel lying on a metal mesh with orifices of 2 mm diameter, and an overlying layer of 15 kg of coarse grained (particle size 5-20 mm) Raney nickel. The catalyst was activated with a 10% solution of caustic soda. The reactor was then charged with an aqueous solution containing 25-30% of caprolactam at the rate of 0.6 m<sup>3</sup>/hr. Hydrogen under an excess pressure of 0.05 kg/cm<sup>2</sup> was supplied at the rate of ~2 m<sup>3</sup>/hr. The quality of the resulting caprolactam was found to improve markedly. The use of the fixed bed nickel catalyst completely precluded the intrusion of the catalyst into the caprolactam solution, thereby eliminating the labor-consuming filtration stage. Toward the end of the tests the unit consumption of the catalyst was 500 g per ton of caprolactam. These findings demonstrate the usefulness of the fixed bed method for the hydrogenation of the impurities, such as carbonyl compounds, present in the aqueous solutions of caprolactam. It is to be expected that the effectiveness of this process will increase still further once the hydrogen pressure is raised to the standard level of 5-6 kg/cm<sup>2</sup>. References: 5 Russian.  
[209-1386]

UDC 542.97+542.952.6+547.315.2

POLYMERIZATION OF BUTADIENE IN THE PRESENCE OF TRIBENZYLTIANIUM

Moscow DOKLADY AKADEMII NAUK SSSR in Russian Vol 251, No 2, 1980 pp 383-385  
manuscript received 20 Aug 79

AFINOGENOVA, L. L., SUZMAN, I. Sh., TINYAKOVA, Ye. I. and DOLGOPLOSK, B. A., Moscow Institute of Petrochemical Synthesis imeni A. V. Topchiyev, USSR Academy of Sciences

[Abstract] The polymerization of butadiene in the presence of tribenzyl titanium was carried out using aromatic hydrocarbons (chiefly *o*-xylene) or a mixture of toluene and heptane. The tribenzyltitanium used was synthesized from tetrabenzyltitanium and lithiummethyl, and all of its titanium existed in the Ti(III) form. The effectiveness and stereospecificity of the resulting polymerization is illustrated by the fact that the resulting polymer has the same structure as that

obtained in the presence of tetrabenzyltitanium, and its vitrification temperature is -42 to -44°C. An attendant investigation of the utilization of titanium-linked benzyl groups during polymerization points to a monotonic decrease in the number of titanium-benzyl links from 3 (in the original tribenzyltitanium) to 0 in the course of the polymerization, with the final product representing a polymeric analogue of tris- $\pi$ -crotyltitanium. Further, the rapid decrease in the viscosity of the reaction mixture following deactivation of the catalyst by methanol points to the formation of centers containing up to three growing polymer chains per titanium atom. Tribenzyltitanium is not as effective as tetrabenzyltitanium in promoting the polymerization of butadiene, because in the latter case the mono-crotyl derivative of Ti(IV) is more stable. Figures 2; references: 3 Russian. [163-1386]

RUBBER AND ELASTOMERS

UDC: 678.065.001.2.01:539.538

INFLUENCE OF INDIVIDUAL DESIGN PARAMETERS OF MOTOR VEHICLE TIRES ON TREAD WEAR

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 45-48 manuscript received  
15 Jun 79

NOVOPOL'SKIY, V. I., TARNOVSKIY, V. N., MAKRAVIN, A. P., Scientific Research Institute of the Tire Industry, Volgograd Polytechnical Institute, Volga Tire Plant

[Abstract] A study was made of the influence of the design of a tire, shape of the tread and physical-mechanical properties of the tread rubber on tread wear rate. Tests were performed under laboratory conditions on a test stand on which the tires roll on a cement drum to better imitate pavement. Automobile tires were tested at 70 km/hr, truck tires at 45 km/hr, with assigned perpendicular, tangential and lateral loads. Both bias-ply and radial-ply tires were tested. Tires with treads in the shape of individual projections were found to wear faster due to the impact nature of the loading with the road than did tires with tread designs which reduced impact loading. In all, changes in tire design, tread pattern and tread rubber formula were found to be able to reduce wear rates by 25-30, 15-17 and 30-32%, respectively. Figures 4, references: 5 Russian.  
[142-6508]

UDC: 678.762.2.023.001.5

STUDY OF THE PROCESS OF MANUFACTURE OF RUBBER MIXTURES BASED ON HIGH VISCOSITY SKD UNDER VARIOUS DEFORMATION CONDITIONS

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 35-38 manuscript received  
9 Feb 79

GOLOLEV, M. A., ZAKHARKIN, O. A., ZAKHAROV, N. D., BEZRUKOV, A. P., Yaroslavl' Polytechnical Institute

[Abstract] Results are presented of studies of the process of manufacture of rubber mixtures for tire treads based on SKD raw rubber, both standard and elevated viscosity, under various deformation conditions. The data produced indicate that high-viscosity-SKD-based rubber mixtures should be manufactured in rubber mixing equipment in which the deformation of the material occurs under conditions approximating pure shear with forced feed of the material into the zone of deformation, as in "transfemix"-type rubber mixers. Figures 5, references: 10 Russian.  
[142-6508]

UDC: 678.044.001.5

INFLUENCE OF N-BENZTHIAZOLINETHIONE DIAMIDOPHOSPHATES ON THE PROPERTIES OF COMMERCIAL RUBBERS MADE OF A COMBINATION OF CRUDE RUBBERS

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 33-35 manuscript received 26 Mar 79

RATNIKOVA, T. V., ZVONKOVA, A. P., SAKAYEV, F. S., KORSHUNOVA, T. N., Leningrad Technological Institute imeni Lensoveta, Volga Rubber Products Plant

[Abstract] A study was made of the influence of N-BDP such as fokaptam E and fokaptam B on the properties of industrial rubbers based on combinations of raw rubbers. These accelerators can initiate heterogeneous vulcanization and therefore increase the probability of producing rubbers with good usage properties. The accelerators were found to increase the fatigue endurance of the vulcanizates, particularly with a sulfur vulcanizing group. Fokaptams also increase the cold strength of the rubbers. Like captax and altax, fokaptams have an isomerizing effect in the process of sulfur vulcanization of rubber. The rubbers produced were found to have over twice the endurance of standard rubbers when tested as the outer coatings of hydraulic brake lines used under northern climate conditions.

References: 8 Russian.

[142-6508]

UDC: (678.762+678.746.22).002.612

PROPERTIES OF ELASTOMERS CONTAINING POLYSTYRENE OF VARYING MOLECULAR MASS

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 18-20 manuscript received 16 Jul 79

KUYKOVA, V. D., YEVREINOV, Yu. V., ARISTOV, O. V., KULEZNEV, V. N.

[Abstract] A study was made of the influence of the molecular mass of polystyrene and its content on the properties of rubber mixtures and vulcanizates based on butadiene methylstyrene rubber (SKMS-30ARK), cis-butadiene rubber (SKD), butyl rubber (BK) and their combinations with polystyrene with molecular masses of  $5.5 \cdot 10^3$  to  $800 \cdot 10^3$ . The rheologic properties of the rubber mixtures produced were studied on a capillary viscosimeter at 130°C. It was found that the strength of the rubbers produced depended not only on the molecular mass of the polystyrene and its content, but also on the method of introduction of the vulcanizing group to the rubber mixture. The curves of strength as a function of polystyrene of various molecular masses all have maxima. However, when the vulcanizing group was introduced to the preliminarily prepared mixture of polymers at 30°C, the rubber had low strength. When the polystyrene was added to the elastomers at 10-15 mass percent, rubbers with high strength and elasticity were produced without changes in the viscosity of the rubber mixture. Figures 3, references: 10 Russian.

[142-6508]

UDC: 678.84:678.048:541.127

INFLUENCE OF THERMAL STABILIZERS ON THE STRUCTURE AND PROPERTIES OF RUBBER MIXTURES BASED ON SILOXANE RUBBERS WITH AEROSIL

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 16-17 manuscript received 19 Dec 78

KAS'YANOVA, I. V., GALIL-OGLY, F. A., Scientific Research Institute of the Rubber Industry

[Abstract] A study was made of the influence of thermal stabilizers on the structure and properties of rubber mixtures based on siloxane raw rubber with fillers. Newly developed thermal stabilizers M-29-PM-75 (a compound of copper), GTSS-50 (technical conditions TU02-1-200-71), lakar (Tu48-1301-45-74) and redoxide as a standard of comparison were used in the experiments. It was found that rubber made of SKTV-1 with M-29-PM-75 thermal stabilizer has high relative elongation and tensile strength, low hardness and elasticity. Rubbers made with lakar and redoxide have greater hardness and less relative elongation at rupture. Thus thermal stabilizers influence the technological properties of rubber mixtures and the strength properties of vulcanizates. Figures 2, references: 5 Russian.  
[142-6508]

UDC: 678.762.3.063.01:539.538:678.048

INFLUENCE OF FIXED AND LOW-MOLECULAR ANTIOXIDANTS ON THE WEAR RATE OF FILLED SKI-3 RUBBER

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 14-15 manuscript received 17 Sep 79

IL'INA, Ye. A., KAVUN, S. M., LYKIN, A. S., TARASOVA, Z. N., National Research Institute of the Tire Industry

[Abstract] A study was made of tread-type rubber made of SKI-3 crude rubber with PM-50 or PM-100 technical carbon, using N-phenyl-N'-isopropyl-p-phenylene diamine and p-nitrosodiphenylamine as antioxidants. The data produced indicate that the influence of inhibitors on the wear resistance of the rubber is more strongly manifested when the rubber contains PM-100 technical carbon. This is probably because the increased activity of the filler decreases the rate of wear of the rubber, thus increasing the contribution of secondary chemical reactions to the process of breakdown. Figures 2, references 4: 3 Russian, 1 Western.  
[142-6508]

UDC: 678.743.41.028:678.043

INFLUENCE OF THE CHEMICAL NATURE OF DIATOMIC PHENOLS AND QUATERNARY AMMONIUM SALTS ON THE PROPERTIES OF VULCANIZATES OF SKF-26 RUBBER

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 10-12 manuscript received 2 Feb 79

LAVROVA, L. N., GILINSKAYA, N. S., SANKINA, G. A., SKOROBOGATOVA, M. S., National Research Institute of the Rubber Industry

[Abstract] A study was made of the influence of the chemical nature of the polyatomic phenol and quaternary ammonium salt on the properties of SKF-26 rubbers. In order to determine the role of the alkyl substituent at the nitrogen atom of quaternary ammonium salts, substances were used which differ only in the nature of this substituent. It was found that hydroquinone, resorcinol and methylresorcinol have high activity for vulcanization of SKF-26. The vulcanizing activity of polyatomic phenols containing the CH group in the o position is not very high. An increase in the length of alkyl substituents in the quaternary ammonium salt improves the resistance of the rubber to thermal aging under stress. Figures 4. [142-6508]

UDC: 678.762.2-136.612.1

INFLUENCE OF THE NATURE OF THE ADDITIONAL MONOMER ON THE PROCESS OF RADICAL POLYMERIZATION OF BUTADIENE IN THE PRESENCE OF SULFUR

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 8-10 manuscript received 4 Jun 79

GARIPOVA, R. A., AVERKO-ANTONOVICH, L. A., Kazan' Institute of Chemical Technology imeni S. M. Kirov

[Abstract] According to the theory of ideal reactivity, the relative reactivity of various radicals in homopolymerization can be estimated by comparing the values of the copolymerization constants  $k_{ab}$  and chain growth constants  $k_{aa}$  of the individual monomers. This article presents the values of  $k_{ab}$  for various radicals in relationship to butadiene and various monomers in relationship to polybutadiene at 60°C. When more active monomers are used, the process of radical copolymerization is slowed to a greater extent by sulfur. All of the bonded sulfur is contained in the polymer in the form of tetrasulfide and disulfide blocks. Figures 2, references 10: 6 Russian, 4 Western. [142-6508]

UDC : 678.84.074:678.4.028

GRAFT COPOLYMERS OF LOW-MOLECULAR METHYLVINYLSILOXANE RUBBERS AND STYRENE

Moscow KAUCHUK I REZINA in Russian No 3, 1980 pp 6-7 manuscript received 25 Jun 79

DAVYDOVA, V. P., KARLIN, A. V., SEVAST'YANOVA, I. V., KOSTYAKOVA, Zh. N., All-Union Scientific Research Institute of Synthetic Rubber imeni S. V. Lebedev

[Abstract] A study was made of the reaction of graft copolymerization of low-molecular  $\alpha$ ,  $\omega$ -methylvinylsiloxane raw rubber and vinyl monomers in a solution with a radical-type initiator, the method of production of the "stirosil"-brand copolymers. It was found that the best properties were produced in a graft copolymer of stirosil type based on methylvinylsiloxane rubber with 1.5 mol.% methylvinylsiloxane links and a content of styrene in the reaction mixture of 45 mass percent. Information is presented on the service life of composites based on stirosil as a function of stannous octoate concentration. References: 6 Russian. [142-6508]

UDC 541.65:539(3+2)

INFLUENCE OF BENZTHIOAZOLINE THIONEDIAMIDE PHOSPHATES ON THE STRUCTURE, MOLECULAR ORIENTATION AND FATIGUE PROPERTIES OF RUBBER

Moscow VYSOKOMOLEKULYARNYYE SOYEDINENIYE in Russian No 4, 1980 pp 919-923  
manuscript received 22 Feb 79

RATNIKOVA, T. V., AKOPYAN, L. A., BARTENEV, G. M., YAKOVLEV, V. I. and GLNAK, A.I.

[Abstract] The influence of organophosphorous accelerants of the benzthioazoline-thionediamide class on the fatigue and strength properties of rubber and on its capacity for molecular orientation in deformation was analyzed to determine the feasibility of using these surfactant compounds as sulfur vulcanization accelerants. The interaction of BDP with rubber, the reaction products that appear during the sulfur vulcanization process, the structure and sulfide content of the vulcanizate cross links were analyzed. The utilization of BDP increases fatigue strength by a factor of 3-6 in relation to the spreading of cracks, and by a factor of 2-3 in relation to alternating bending with a 10-30° reduction of heating. Figures 2; tables 1; references 18: 17 Russian, 1 Western.  
[157-7872]

UDC 542.97:547.315.2

BUTADIENE CYCLODIMERIZATION IN PRESENCE OF SECONDARY AMINES CATALYZED BY NICKEL COMPLEXES

Moscow IZVESTIYA AKADEMII NAUK SSSR, SERIYA KHMICHESKAYA in Russian No 6, Jun 80 pp 1324-1327 manuscript received 26 Mar 79

DZHEMILEV, U. M., FAKHRETDINOV, R. N., and TELIN, A. G., et al., Institute of Chemistry, Bashkir Branch, USSR Academy of Sciences, Ufa

[Abstract] The telomerization of butadiene with various amines in presence of nickel complexes activated with chiral phosphates was studied. The reactions, which lasted from 140 to 840 hrs, were carried out in sealed ampoules at -8°C to -60°C using a threefold excess of diene. To a solution of 0.002 mole of  $\text{Ni}(\text{Acac})_2$ , 0.006 mole phosphite and 10 ml butadiene in 20 ml benzene, 0.008 mole of  $\text{Al}(\text{C}_2\text{H}_5)_3$  was added in an atmosphere of argon at -5°C; the mixture was stirred for 20 min and the solution was placed in a glass ampoule containing 0.6 mole butadiene and 0.2 mole amine. The sealed ampoule was kept for different lengths of times at various temperatures to yield 4-ethylcyclohexene-1 and 4-vinylcyclohexene-1.

Figure 1, references 7: 6 Russian, 1 Western.

[210-7813]

UDC 678.742.2-945.254.32.004.12.001.5

OPTIMAL HIGH PRESSURE POLYETHYLENE FOR PRODUCTION OF CHLOROSULFOPOLYETHYLENE ELASTOMERS

Moscow KAUCHUK I REZINA in Russian No 1, 1980 pp 5-8 manuscript received 15 May 79

RONKIN, G. M., KOROTYANSKIY, M. A., GERSHENOVICH, A. I., DZHAGATSPANYAN, R. V.

[Abstract] Results are presented of a comparative study of the properties of chlorosulfonated polyethylene produced from various types of high pressure polyethylene with a melt flow index of 1.21-3.28 g/10 minutes and a mean-viscosity molecular mass of  $11.2-53.6 \cdot 10^3$ . The molecular mass was determined by the characteristic viscosity at 80°C. The degree of branching of the PE chains was determined by IR spectroscopy; crystallinity was determined on a diffractometer. The results of the measurements are presented in tabular form. It is found that the optimal properties are produced by the use of high pressure polyethylene with a mean-viscosity molecular mass of 25,000-30,000 and narrow MMR (20,000-25,000). Figures 3, references 9: 4 Russian, 5 Western.

[134-6508]

UDC 678.684.82.028:678.643'42'5

INFLUENCE OF E-40 EPOXY RESIN ON THE PROCESS OF VULCANIZATION OF POLYSULFIDE OLIGOMER AND PROPERTIES OF VULCANIZATES

Moscow KAUCHUK I REZINA in Russian No 1, 1980 pp 12-15 manuscript received 21 Jan 77

MUKHUTDINOVA, T. Z., SHAKHMAYEVA, A. K., GABDRAKHMANOV, F. G., SATTAROVA, V. M., Kazan' Branch, All-Union Scientific Research Institute of Synthetic Rubber imeni S. V. Lebedev

[Abstract] Results are presented of studies of the influence of E-40 epoxy resin on the process of vulcanization of polysulfide oligomer and the properties of its vulcanizates. 0-20 mass parts of epoxy resin, manganese-dioxide-based vulcanizing paste No. 9 with dibutylphthalate, diphenylguanidine and PM-15 technical carbon were added to an oligomer based on di-( $\beta$ -chloroethyl)formal. The composition was vulcanized for 24 hours at room temperature and 24 hours at 70 C. The kinetics of vulcanization of the composition were studied by periodic measurement of viscosity. In speci. as without epoxy resin, 92-94% of the polysulfide oligomer was included in the vulcanization grid. Introduction of the epoxy resin had a negative influence on the degree of vulcanization of the polysulfide oligomer: with 20 mass parts epoxy resin, only 53% of the polysulfide oligomer was vulcanized, the remaining portion merely plasticizing the vulcanizate. Even acetone was capable of extracting up to 13% of the oligomer and 60-83% of the resin from vulcanizate containing 20 mass parts epoxy resin. The reason for the decreased degree of structuring of the polysulfide oligomer in the presence of epoxy resin is apparently the breaking of chains as a result of partial interaction of the terminal thiol groups with the epoxy resin groups. New accelerators and vulcanization conditions must therefore be developed for the use of polysulfide oligomer in mixtures with epoxy resin to produce materials with good mechanical properties. Figures 3; references 10: 8 Russian, 2 Western.

[134-6508]

UDC 678.4.063.019.347.12:531.783

PREDICTION OF THE EFFECTIVENESS OF RUBBERS IN LIQUID NONCOMBUSTIBLE MEDIA

Moscow KAUCHUK I REZINA in Russian No 1, 1980 pp 22-27 manuscript received 23 Sep 77

AKOPYAN, L. A., ZOBINA, M. V., BERDENIKOV, A. I., BARTENEV, G. M., Leningrad Branch of Scientific Research Institute of the Rubber Industry, Institute of Physical Chemistry, USSR Academy of Sciences

[Abstract] The influence of the nonflammable hydraulic fluid substitute PGV, an aqueous solution of polyethylene glycol and glycerine plus additives, on the rate of physical and chemical relaxation processes in rubbers was studied by the method of relaxation spectrometry. Tests were performed on SKF-32, SKN-18 and SKN-26 rubbers. Stress relaxation in the rubbers was measured 5 minutes after heating of

the specimens to 20-110 C, at 20% uniaxial compressive deformation. The time required to reach 60% of the initial modulus of elasticity in liquid PGV and in air is predicted. The results indicate that the service life of the rubbers tested in PGV would be at least equal to their service life in air. More accurate methods of prediction of the rate of decrease of elasticity modulus would allow rubber products in contact with PGV substitute hydraulic fluid to be used for longer periods of time before routine replacement. Figures 3, references: 10 Russian.  
[134-6508]

UDC: 678.762.3:678.048.001.5

#### INFLUENCE OF CONCENTRATION OF DFFD ANTIOXIDANT ON THE STABILITY OF SKI-3 RAW RUBBER NOT CONTAINING NEOSONE D

Moscow KAUCHUK I REZINA in Russian No 1, 1980 pp 35-37 manuscript received 5 Mar 79

L'VOV, Yu. A., SENDERSKAYA, Ye. Ye., PANINA, I. S., PIOTROVSKIY, K. B., All-Union Scientific Research Institute of Synthetic Rubber imeni S. V. Lebedev

[Abstract] A study was performed to determine the optimal concentration of DFFD (*N*, *N'*-diphenyl-p-phenylenediamine) in SKI-3 raw rubber stabilized without neosone D (phenyl- $\beta$ -ethylamine), and to determine the stability of experimental batches of SKI-3 rubber with DFFD antioxidant in the process of thermal oxidation and long-term storage. The experiments showed that the greatest stability was achieved with a DFFD content of about 0.1 mass percent. Higher content of DFFD accelerate destruction both under conditions of thermal oxidation at 150 C and during thermo-mechanical working in rolls at 125 C. DFFD in quantities of not over 0.3 mass percent also assures high stability in long-term storage (tests extended up to 36 months). Figures 2, references: 3 Russian.

[134-6508]

UDC: 678.4.023.334:(678.762.2-134.352;  
678.743.41):678.46:62-492.2

#### MANUFACTURE OF HIGH-VISCOSITY RUBBER MIXTURES BASED ON SKN-40 AND SKF-32 WITH GROUND VULCANIZATE IN A CONTINUOUS RUBBER MIXER

Moscow KAUCHUK I REZINA in Russian No 1, 1980 pp 40-42 manuscript received 3 Nov 78

VORONIN, A. D., ZAKHARKIN, O. A., ZAKHAROV, N. D., TAMARKIN, V. F., Yaroslavl' Polytechnical Institute, Scientific Research Institute of the Rubber Industry

[Abstract] A study was made of the influence of the content of finely ground vulcanizate on the technological parameters of the continuous process of manufacture of highly viscous rubber mixtures based on SKN-40 and SKF-32 raw rubber, and on the properties of the mixtures and the rubbers. When the content of finely ground vulcanizate in the system is over 30 mass parts, the effectiveness of

dispersing mixing decreases, manifested as a decrease of electric power consumption and crumbling of the mixtures, particularly in SKF-32-based systems. The change in viscosity and cohesion strength of mixtures upon addition of the powders is illustrated. Introduction of up 30 mass parts to SKN-40-based rubber does not greatly change the mechanical properties of the rubber. Addition of over 20 mass parts to SKF-32-based rubber causes a tendency toward decreased tearing resistance, tensile strength and relative elongation. Figures 4, references: 7 Russian.  
[134-6508]

#### FLAMMABLE AND TOXIC PROPERTIES OF INGREDIENTS

Moscow KAUCHUK I REZINA in Russian No 1, 1980 p 62

[Abstract] Flammable and toxic properties are listed for oleic acid and diaphen FP. General characteristics are listed, then it is noted that materials containing compounds of metals of variable valence may cause fire or explosion if they come in contact with oleic acid, while diaphen FP is toxic, MPC 2 mg/m<sup>3</sup>, requiring the use of skin protection, protection of mucosa and the organs of respiration. Preferred methods of fire fighting and storage conditions are noted.

[134-6508]

## MISCELLANEOUS

UDC 347.77.001.2

### COMPREHENSIVE PATENT SEARCHES AS A FACTOR IN ENHANCING THE EFFECTIVENESS OF SCIENTIFIC RESEARCH

Aks-Ata VESTNIK AKADEMII NAUK KAZAKHSKOY SSR in Russian No 2, 1980 pp 31-35

SOKOL'SKIY, D. V., ZAVDROCHINA, N. A. and VOSYAKOVA, Ye. N.

[Abstract] Patent searches are of major importance to guiding the course of scientific research to develop competitive and patentable innovations. The Institute of Organic Catalysis and Electrocatalysis, Kazakh SSR Academy of Sciences, is chiefly doing research in such fields as catalysts for hydration, hydrogenation, and oxidation processes; synthesis of acetylene- and ethylene-based materials; homogeneous and heterogeneous activation of molecules of nitrogen, hydrogen, and carbon monoxide; techniques for obtaining pure and superpure metals; and development of new types of fuel cells and electrodes. The Institute has enlisted the assistance of the Alma-Ata Affiliate of the All-Union Center for Patent Services (VTSPU). This collaboration helps the institute to avoid research duplication and to keep track of worldwide developments in its fields. Currently the Institute is working on 13 research topics and 20 contracts. In 3 years alone it has been granted 76 Soviet patents for its innovations, plus 19 foreign patents. For example, the Institute's scientists have developed in collaboration with the Chinkent Lead plant a new technology for the production of superpure lead for which the Institute was awarded gold medals at expositions. References: 10 Russian.  
[191-1386]

UDC 541.127

### EFFECT OF A MAGNETIC FIELD ON OXIDATION OF CHOLESTERIC LIQUID CRYSTALS

Yerevan ARMYANSKIY KHIMICHESKIY ZHURNAL in Russian No 4, 1980 submitted 27 Oct 77 signed to press 30 Apr 80 pp 329-331

PARYAN, S. V., VARDANYAN, R. I. and SARKISYAN, A. Ts., Goris Physico-Technical Department of the Armenian Branch of the All-Union Scientific Research Institute of Chemical Reagents and Ultrapure Chemical Substances, Goris Branch of the Armenian Pedagogical Institute imeni Kh. Abovyan

[Abstract] A study of cholestrylpropionate, cholestrylbenzoate and cholestryllaurate with the use of a radiospectrometer electromagnet, with rate of oxidation

determined by oxygen absorption using a manometric device in a diffusion regime and comparison of results with cholesteric liquid crystals oxidation in the absence of a magnetic field, showed the effect of the magnetic field on the rate of oxidation reaction under the experimental conditions to be much greater than the rates predicted theoretically. It was assumed that, in addition to the effect of the magnetic field on the direction of S  $\leftrightarrow$  T transitions in radical pairs, the field also affects some other still unknown mechanism. References: 8; figure 1.  
[213-2791]

CSD: 1841

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